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(54) Method for decontamination of a chamber used in vacuum processes for deposition, etching and/or growth of high purity films, particularly applicable to semiconductor technology.

(57) After introducing the products concerned with the process into the chamber and after creating a vacuum in said chamber, said chamber is decontaminated by a series of inflows of non-contaminating gas and subsequent emptying operations.

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EUROPEAN SEARCH REPORT

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EP 87 20 2219

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
A	JOURNAL OF THE ELECTROCHEMICAL SOCIETY, vol. 132, no. 3, March 1985, pages 642-648, Manchester, New Hampshire, US; P. LIAW et al.: "Epitaxial growth and characterization of beta-SiC thin films" * Page 643, right-hand column, lines 16,17 *	1-5	C 23 C 16/44 C 30 B 23/02 C 30 B 25/08
A	PATENT ABSTRACTS OF JAPAN, vol. 10, no. 283 (E-440)[2339], 26th September 1986; & JP-A-61 102 048 (NEC CORP.) 20-05-1986 * Abstract *	1,3,5	
A	PATENT ABSTRACTS OF JAPAN, vol. 7, no. 123 (E-178)[1268], 27th May 1983; & JP-A-58 40 841 (NIPPON DENKI K.K.) 09-03-1983	1,3,5	
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
			C 23 C C 30 B

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	14-03-1988	ELSEN D.B.A.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
O : non-written disclosure
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"METHOD FOR DECONTAMINATION OF A CHAMBER USED IN VACUUM PROCESSES FOR DEPOSITION, ETCHING AND/OR GROWTH OF HIGH PURITY FILMS, PARTICULARLY APPLICABLE TO SEMICONDUCTOR TECHNOLOGY"

This invention concerns a method for decontaminating chambers used in vacuum processes for deposition, etching or growth of high purity films, particularly applicable in semiconductor technology and, specifically, in processes for the deposition, etching and/or growth of films on semiconductor wafers.

In the technology of semiconductor devices, use is preferably made, for deposition, etching or growth of films, of vacuum processes in order to reduce the presence of contaminating substances in the chambers where the processes take place, and consequently to improve the chemico-physical and stoichiometric characteristics of the films obtained. Use is made in particular of systems such as Low Pressure Chemical Vapor Deposition (LPCVD), Plasma Enhanced CVD (PECVD), evaporation, sputtering, Reactive Ion Etching (RIE), systems for etching in plasma, etc..

Known procedure, relating to a vacuum process for deposition or etching of films, generally begins with creation of a vacuum in the chamber until a certain minimum pressure level is reached (about 1-25 milliTorr), followed by decontamination of the chamber by allowing non-contaminating gas to flow in at low pressure (e.g. 200 millTorr) for a previously set time (between 10 and 40 minutes). Then, having once more brought the vacuum down to a certain minimum value, the deposition or the etching processes begins. Although this method appreciably lessens the presence of undesired gases in the chamber (such as H, H_2, CO_2, OH, H_2O, etc.) it however fails to reduce it below certain limits.

The method of decontamination subject of this present invention, aims at appreciably reducing - leaving un changed the overall decontaminating time - the presence of most of the contaminants below the limit values rereferred to above, securiting advantages as far as concerns the chemico-physical and stoichiometric characteristics of the products obtained thereby, such advantages being, for example, reduction of current leakage in insulating films, more efficient film gettering, red··ction of the non-stoichiometric zones in the films, more uniform etching, greater possibility of reproducing the effects obtained by etching the films, etc..

For this purpose, after placing the products in the chamber for processing and before that is started, the method requires a vacuum, to a certain minimum level of pressure, to be created inside the chamber (hereinafter, for simplicity, this first step will be called "pumping"), and then its decontamination by flowing in non-contaminating gas at a pressure suitably higher than the above minimum level (hereinafter, for simplicity, this second step will be called "purging"), this method being characterized in that the flow of non-contaminating gas is intermittent, decontamination being achieved by a series of purgings, each purging being followed by a pumping.

These and other characteristics of the invention will be made clearer by the description that follows and by the attached drawings illustrating an example of its realization of a non-limiting kind, in which the various figures show:

Fig.1: diagram of a known process of deposition or etching of films in a vacuum;

Fig.2: diagram of a process of deposition or etching of films according to the invention;

Fig.3: diagram of a unit functioning according to the process in Fig.2;

Figs.4A and 4B: diagrams showing levels of contamination noted at the end of two decontamination processes executed by the known technique;

Figs.5A and 5B: diagrams showing levels of contamination noted at the end of two decontamination processes executed according to the invention.

The diagram in Fig.1 concerns an example of an already known process of deposition or etching of a film. According to this example, after the wafers have been placed in the chamber, at atmospheric pressure, where the process takes place, (said chamber consisting, for example, of a cylindrical body with quartz walls, heated from the outside), a vacuum is created by rotary pumps assisted if necessary by Roots pumps. When pressure has been reduced to about 25 mTorr, in roughly 10 minutes, the decontamination process begins (lasting for about 50 minutes), and, during this, nitrogen is flowed into the chamber. A system that regulates the opening of the N_2 outflow valves ensures that when pressure has risen to about 200 mTorr, it remains constant at that level for about 45 minutes. When this time has passed, the N_2 outflow valve is closed and the pump for creating and maintaining the vacuum reduces pressure once more. Pressure having fallen to about 12 mTorr, the valves controlling inflow of the gases used for deposition or etching of the film are opened and the process begins.

The diagram in Fig.2 illustrates an example of a deposition or etching process according to the invention.

In said process the initial stage consisting of placing wafers in the chamber and subsequent creation of a vacuum therein, is exactly the same as that for Fig.1. When pressure has fallen to about 25 mTorr, the decontamination process starts with flowing in N_2, lasting still about 50 minutes. Contrary to the preceding case, however, the N_2 inflow valve is alternately opened and closed at regular time intervals of about 2 minutes, giving rise to a series of 13 purgings and subsequent pumpings, and to a pressure graph rising and falling beween about 200 mTorr and 25 mTorr (except for the last cycles of the series when minimum pressure drops to about 12 mTorr). The decontamination process is therefore followed by the process of deposition and/or etching of a film similary to what happens in the case of Fig.1.

Fig.3 gives a schematic example of a layout of a low pressure system for deposition or etching of films according to the invention.

The system comprises a chamber C in which the deposition process takes place, a pump P for creating and maintaining low pressure in C, a nitrogen container X, containers Y and Z for the reagent gases used during the deposition or etching process of the film, valves V1, V2, V3 and V4 for regulating gas flows, valves V5 and V6 for regulating gas flow out of chamber C, a gauge M for measuring pressure inside C and, finally a unit R for controlling and regulating gas flows and pressure in C. When the process starts wafers are already inside C, the pump P is working and door D to the chamber is closed as are all the valves V1, V2, ...,V6. Valve 6, a half-gate valve, is then opened and this connects the chamber C to the pump; said valve is kept open for about 5 minutes while the pressure falls from 760 Torr to about 500 mTorr. At this point valve V6 is closed and valve 5 (gate valve) opened enabling a stronger vacum to be obtained. After 5 more minutes when the pressure has fallen to about 25 mTorr, leaving valve V5 still open, valve V1 is opened allowing N_2 to enter the chamber. After about 2 minutes when pressure has risen to about 200 mTorr (this value coinciding with the pressure chosen for carrying out the next deposition or the next etching), valve V1 is closed again and kept closed for another 2 minutes until pressure has once more dropped to the minimum level (about 25 mTorr). This first purging and pumping is followed by 12 purging and pumping cycles (all done in a substantially similar manner except for the last pumping when pressure falls to a minimum of about 12 mTorr), after which the decontamination process is completed and a start is made with the process of deposition or etching by opening valves V2, V4 and V3.

To enable an evaluation to be made of the appreciable reduction of contaminants allowed by the invention, in Figs. 4A and 4B will be seen diagrams showing the concentrations recorded by a mass spectrometer (of the Residual Gas Analysis - RGA - type) after a decontamination process of the conventional kind, respectively carried out in a chamber for deposition of a polycrystal silicon film (Fig.4A) and in a chamber for deposition of a silicon nitride film (Fig.4B).

Figs. 5A and 5B however show diagrams relating to the concentrations found in those same chambers when - other conditions and particularly the time taken being equal -decontamination is done according to the present invention (Fig.5A) relating to the chamber for deposition of the polycrystal silicon film, and 5B to the chamber for deposition of silicon nitride film). In the case of Fig.5A the diagram brings out the decrease D in concentration of the various polluting substances, these values being:

$D(H) = 26.2\%$; $D(H_2) = 22.9\%$; $D(O_2) = 0\%$; $D(OH) = 27.8\%$; $D(H_2O) = 27.6\%$; $D(CO_2) = 0\%$

while in the case of Fig.5B there was a decrease in the following values:

$D(H) = 24\%$; $D(H_2) = 25\%$; $D(O_2) = 0\%$; $D(OH) = 27.9\%$; $D(H_2O) = 29.8\%$; $D(CO_2) = 0\%$.

In the example of realization described above reference has been made to a particular low pressure system and to the use of nitrogen in the decontamination process. The same method can however be applied both in connection with all the possible gases normally used for the purpose, and in connection with the various vacuum systems operating at pressures in the region of milliTorr (LPCVD,PECVD, evaporation, sputtering, RIE, for etching in plasma systems, etc.) or at pressures in the region of Torr (systems for the growth of epitaxial layers of silicon, etc.).

Further, this example is not be considered as limited to semiconductor technology as it is obviously applicable to all the vacuum processes that concern deposition, etching or growth of high purity films.

Claims

1. Method for decontamination of a chamber used in vacuum processes for deposition, etching and/ or growth of high purity films, to be applied in particular to semiconductor technology, which requires, after the introduction into the chamber of the products to be subjected to the process and before said process begins, the creation of a vacuum in said chamber until a certain minimum pressure value is reached (called hereinafter for simplicity "pumping") and then its decontamination by flowing in non-contaminating gas at a pressure suitably higher than the above minimum value (this

operation called hereinafter for simplicity "purging"), characterized in that the non-contaminating gas flows in intermittently, decontamination being realized by a series of purgings each of which is followed by a pumping.

2. Method as in claim 1 characterized in that each purging and each pumping operation in the above series is started as soon as pressure inside the chamber has reached a certain minimum and respectively maximum value.

3. Plant for vacuum processes of deposition, etching or growth of high purity films to be applied particularly to semiconductor technology which includes first means capable, following introduction into the chamber of the products to be subjected to the process and before said process starts, of creating a vacuum in said chamber down to a certain minimum value of pressure (called hereinafter for simplicity "pumping") and also second means capable of subsequently decontaminating the chamber by a flow of non-contaminating gas at a value of pressure suitably higher compared with the minimum value referred to above (called hereinafter for simplicity "purging"), characterized in that said first and second means cause the flow of non-contaminating gas to be intermittent, decontamination being realized by a series of purgings each one followed by a pumping.

4. Plant as in claim 3 characterized in that said first and second means regulate the above mentioned series of purgings and subsequent pumpings in such a way that each purging and pumping operation begins as soon as pressure inside the chamber has reached a certain minimum and respectively maximum value.

5. Products obtained according to a manufacturing process the phases of which include deposition, etching or growth of high purity films and decontamination of the chamber used for this purpose by means of the method described in the preceding claim 1.

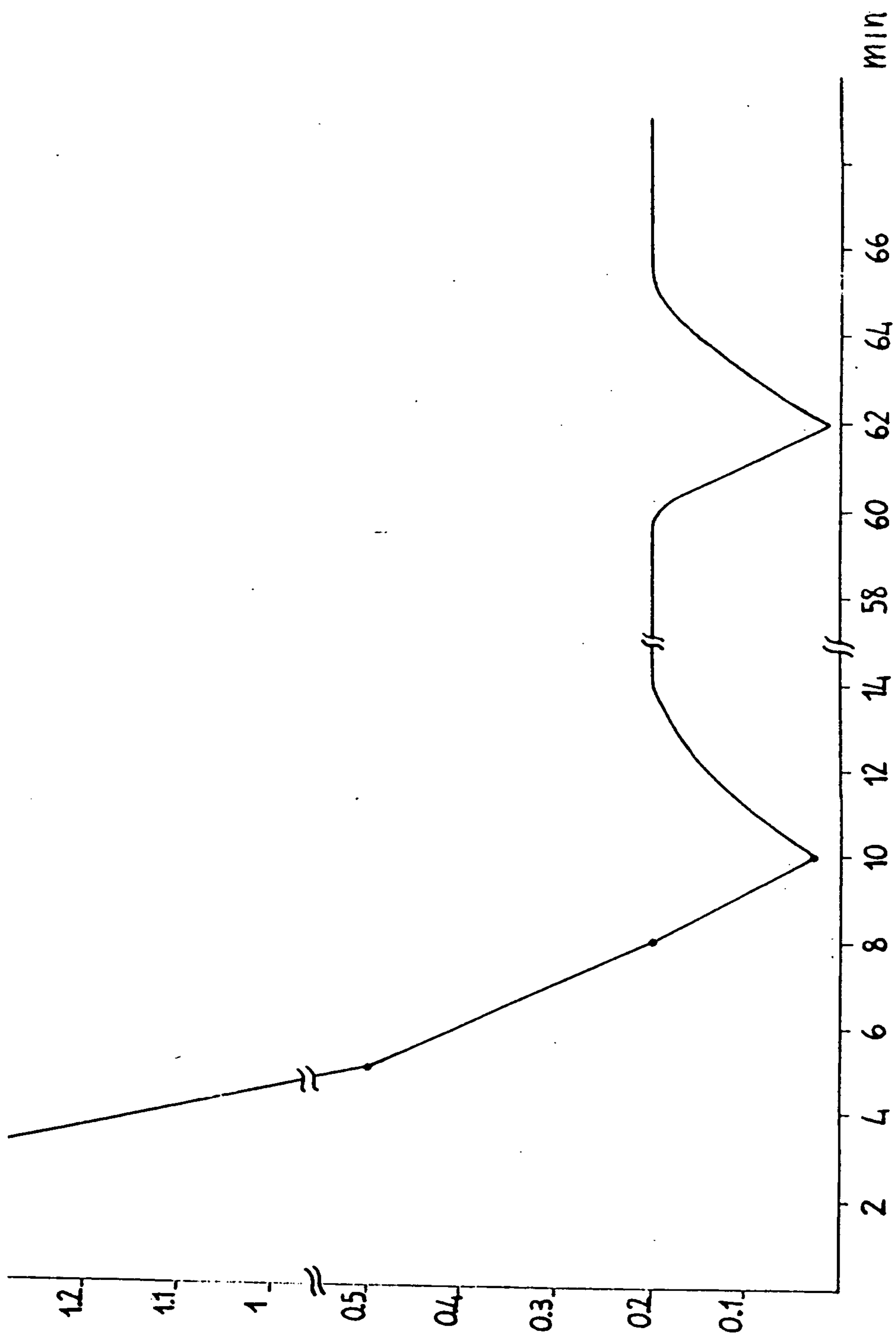

FIG.1

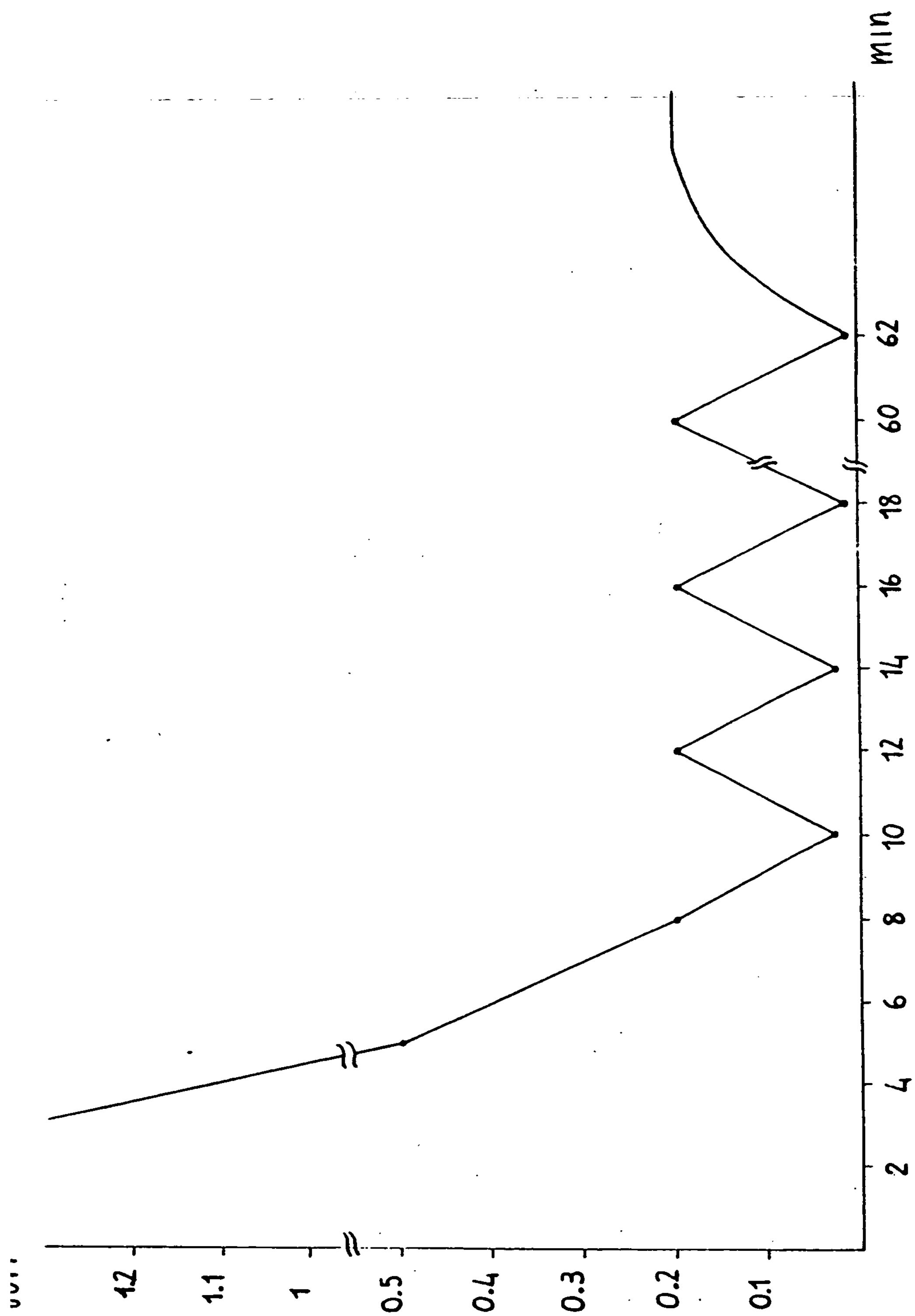

FIG.2

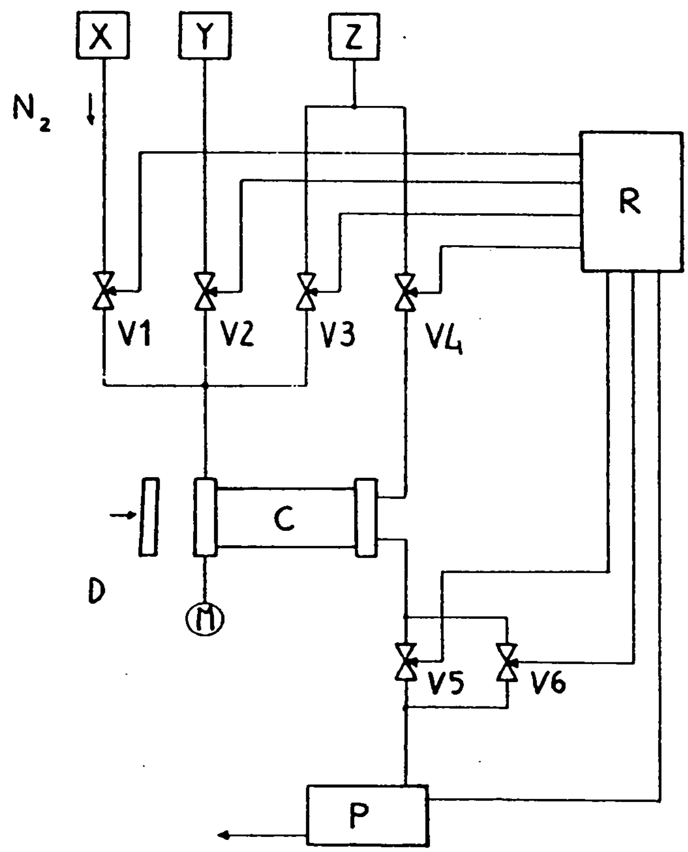

FIG. 3

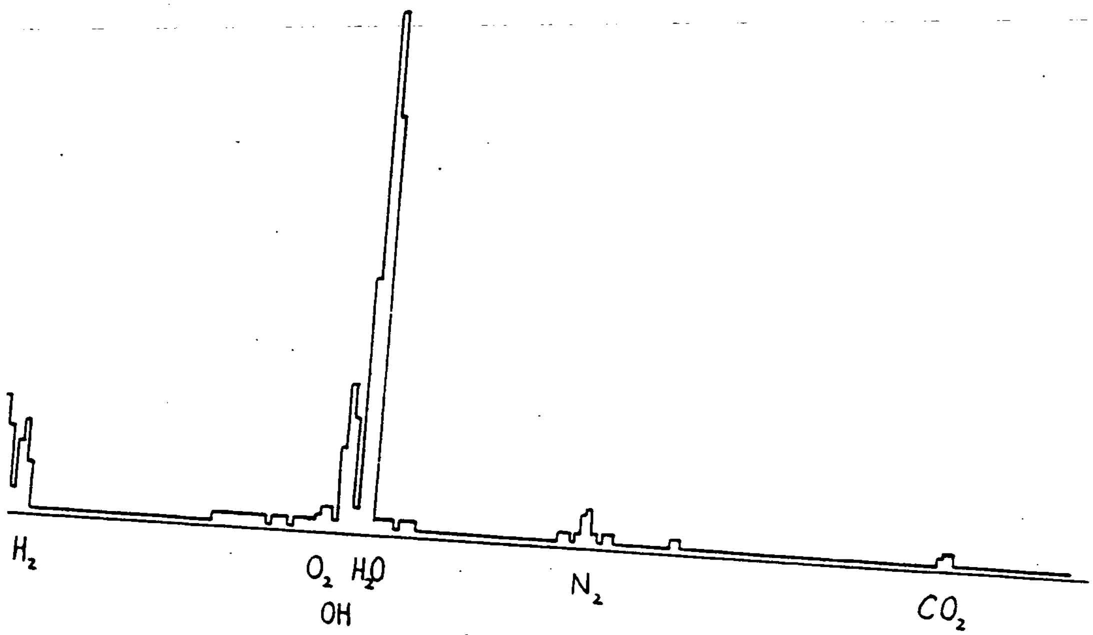

FIG. 4A

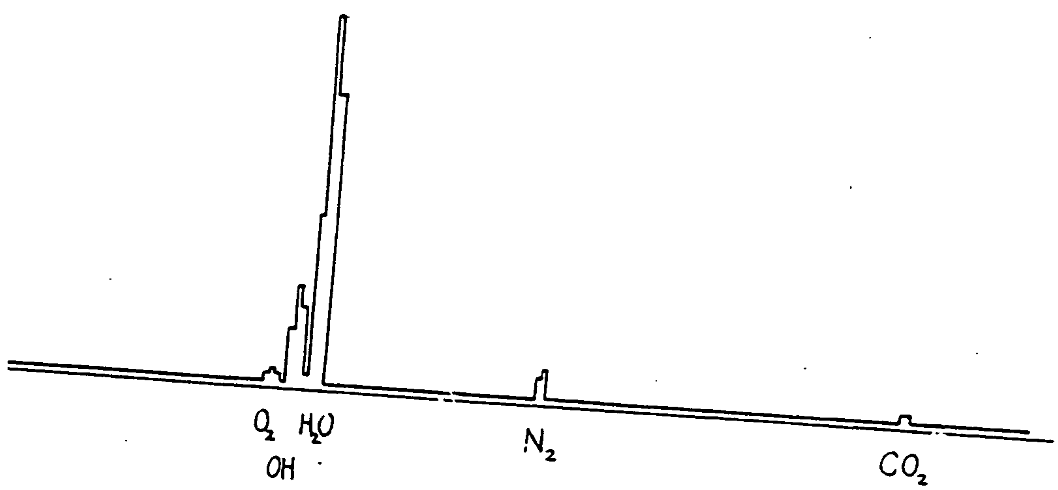

FIG. 5A

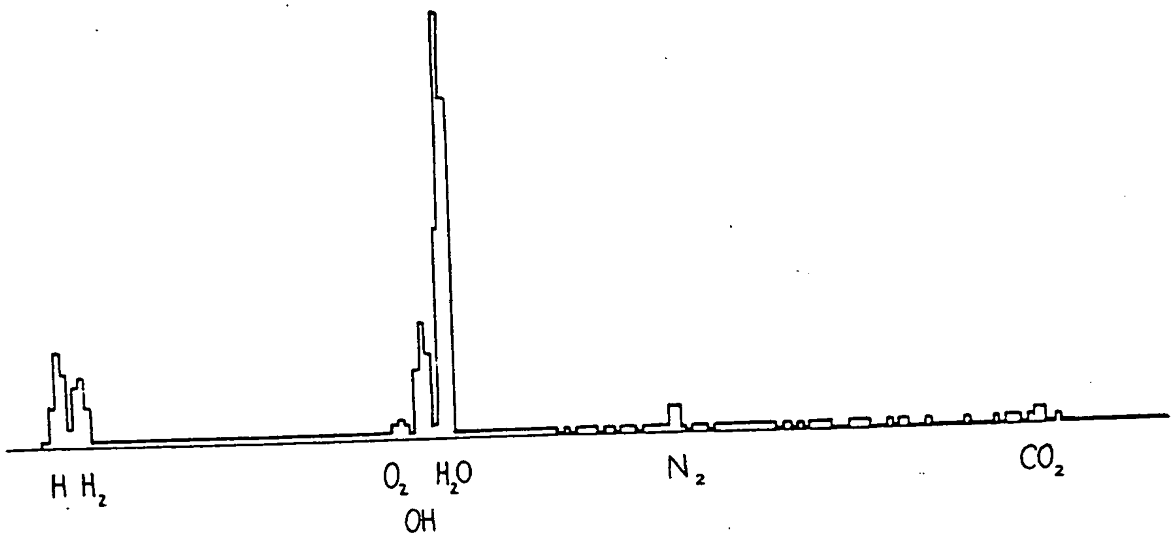

FIG. 4B

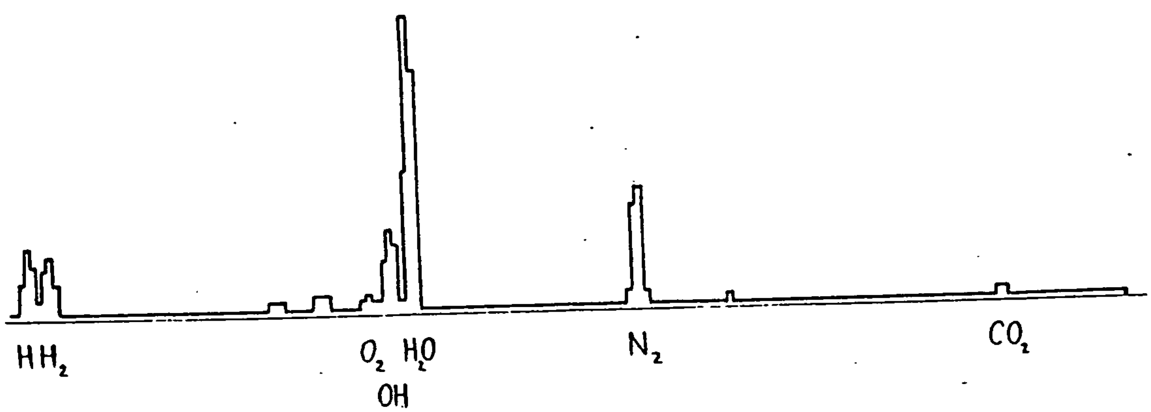

FIG. 5B

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(54) GAS SUPPLY PIPELINE SYSTEM FOR PROCESS EQUIPMENT.

(57) A gas supply pipeline system for process equipment, adapted to supply at least two kinds of process gas, and provided with at least two valves (135, 136, 139, 140) installed in each of independent flow passages formed between process gas supply pipelines and a purge gas supply pipe line, and at least two valves (137, 138, 141, 142) installed in each of a plurality of flow passages formed between the process gas supply pipelines and the pipelines in the process equipment. Each of the process gas supply pipelines and each of the pipelines in th proc ss equipment can be purged and vacuumed by at l ast two valves in each of these flow passages that are opened or closed independently, and the stagnation of a gas in the pipelines not in use can be prevented by introducing a purge gas constantly thereinto.

EP 0 379 594 A1

SPECIFICATION

A GAS SUPPLY PIPING DEVICE FOR A PROCESS APPARATUS

TECHNICAL FIELD

The present invention relates to a piping device for supplying process gas of dry etching process for various thin film formations or fine patterns, and particularly relates to a gas supply piping device for a process apparatus, which makes possible high quality film formations and high quality etching.

BACKGROUND TECHNOLOGY

In recent years, in the dry etching process for high quality thin film formations or fine patterns, the ultrapurification of the process atmosphere, i.e. the technique of supplying ultrapure gases to process apparatuses has become very important.

For instance, in the case of semi-conductor devices, to increase the integrity of integrated circuits, the dimension of unit elements has diminished year after year; in order to practicalize semi-conductor devices of 1 micron to the size of submicron, or even smaller than 0.5 micron, research and development are being actively undertaken. The manufacture of this kind of semi-conductor device is carried out by means of repeating formations of thin films and etching these thin films into specific circuit patterns. Then, this kind of process is usually done by putting the silicon wafer

the purifying device to the semi-conductor manufacture device. Therefore, through the development and improvement of storage tanks, purifying devices, and piping materials, suppyling ultrapure gases to semi-conductor manufacturing devices has become possible. (Ohmi Tadahiro. "A challenge to ppt--a challenge to the concentration of impurities approximating ppt by the gas piping system for semi-conductors." Nikkei Microdevice. July 1987, pp. 98-119.) On the other hand, for special material gases, as careful attention is needed in handling them, the quantity used is very small in comparison to normal gases; therefore, gases in the cylinder are introduced by pressure to the semi-conductor manufacturing device through a cylinder-cabinet piping device.

Up to now, the most serious problem in the ultrapurification of gases supplied from the cylinder through the cylinder-cabinet piping device is the dirt inside the cylinder itself, the existence of large external leakage in the joint of the cylinder-valve and the cylinder, as well as pollution caused by productive absorbing gases, which are engendered because the inside of the cylinder is not accessible to cleaning. However, this problem is also almost overcome by treating the inside to become a specular surface of no processing deterior layer by means of complex electropolish, and the development of cylinder-valves having

mixed from the various process gases before transporting them to the process device. Under this circumstance, when selecting and mixing process gases, if some process gas is left behind in the process gas supply and control pipeline, then replacement of process gas wil not take place in that part, hence reducing the degree of purity of the process gas to be supplied to the process device. Besides, although there is no problem concerning the process gas supply and control pipeline when it is used to supply process gas to the process device, the process gas supply and control pipeline is oftentimes in a state of sealing in gases when it is not used to supply process gas; therefore, it will be polluted by the gas chiefly released from water moisture in the inside of the pipeline; and when the process gas supply and control pipeline is used again, this will become the reason for the reduction in the degree of purity of the process gas supplied.

In this way, if the flowing of gas stops and residual gas exists in the gas piping system, it occurs to pollute the piping system and to reduce the purity of the supplied gas. As a result, the process will be tremendously affected.

For instance, the most recently developed DC-RF coupled mode bias sputtering device can also obtain an excellent Al thin film of specular surface with entirely no hillock, even

growth and epitaxial growth can be seen. That is, the epitaxial growth of Si on clean Si surface can also be obtained; the film formation of polysilicon above SiO_2 can be suppressed to very few (Murota Junichi, Namamura Naoto, Kato Manabu, Mikoshiba Nobuo, and Ohmi Tadahiro. "An Ultracleaning CVD Technique Having High Selectivity." The Sixth ULSI Ultracleaning Technology--Symposium. A Collection of Drafts "High Performance Process Techniques III." Jan. 1988, pp. 215-226).

Fig. 31 (a) through (c) is a most preferred embodiment of the known gas supply pipeline of the process apparatus which supplies a number of process gases to the process apparatus.

A brief description of the above is given with reference to Fig. 31 (a). For simplicity sake, Fig. 31 (a) shows three kinds of process gas are supplied to the process gas supply piping device for the process apparatus. In Fig. 31 (a), 601 is the reaction chamber of the process apparatus, which is connected with the vacuum exhaust device. 608, 609, 610, 614, 615, 616, 617, 618, 619, 629, 630, 631, 632, 633, 634, 635, 636, 637, 643, 644, 648, 649, 650, 652, 653, 654, 658, 659, and 663 are stop valves; among these, 614 and 617, 615 and 618, 616 and 619, as well as 653 and 654 are respectively 3-way dual valves integrating two valves into one. 602, 603, and 604 are process gas supply

shown in Fig. 31 (a) are described with reference to Fig. 31 (b) through (f). Here, the supply of process gas from the process gas supply piping line 602 to the reaction chamber 601 is used as an example, and its operation is separately explained in points (1) to (5).

(1) When the Device Stops

As a general rule, when the process device is not used for the process gas, as shown in Fig. 31 (b), and when stop valves 608, 609, 610, 614, 615, 616, 617, 618, 619, 629, 630, 631, 635, 636, 637, 643, 644, 648, 649, 650, 654, 658, 659 and 663 are all in an open state, and stop valves 632, 633, 634, 652 and 653 are all in a close state, the purge gas (e.g. Ar) passes from the process gas supply piping lines 602, 603, and 604 through needle valves 656 and 661 for flow regulation to the front of the reaction chamber of the process device; and all this time the purge gas inside the piping system is constantly flowing. The thick lines represent the flow of the gas.

(2) When Process Gas Is Substituted for Purge Gas

Next, in order to supply ultrapure process gas to the process apparatus, the operation of substituting the residual purge gas (e.g. Ar gas) in the supply piping system with process gas is carried out. First of all, from the state as shown in Fig. 31 (b), close valves 658, 663, 654, 656, 659, and 661, then close valves 635, 636, 637, 649, and

the supply pressure and flow of process gas by means of the pressure regulator 611 and the mass flow controller 620 and supply the process gas to the reaction chamber 601. Then close valve 652, open valves 649, 650, 659, 661, and 663, and begin anew the purging of piping lines 603 and 604, which have not supplied process gas. This state is as shown in Fig. 31 (e).

(4) Stopping the Supply of Process Gas

Following, the method of stopping the supply of process gas is described. This operation is similar to that of the supplying process gas; not that process gas will substitute for purge gas, but that purge gas (e.g. Ar) will substitute for process gas. The supply of purge gas and the vacuum exhaust discharge in the piping lines are usually repeated more than five times in this operation. Then, close valves 608, 614, 617, 629, 632, 635, 643, 644, 648, 652, and 653, and supply purge gas from the process gas supply piping line 602. Next, open valves 608, 614, 617, 629, 635, 643, 644, 648, 654, 656, 658, 659, 661, and 663, and begin purging (Fig. 31 (f)). When purging starts again from the system of piping lines 603 and 604, the state shown in Fig. 31 (b) is obtained.

However, although, in the apparatus shown in Fig. 31 (a), for instance, when process gas from the process gas supply pipeline 602 is supplied or checked, the substitution

of the piping system, hence becoming a big problem for the process apparatus of the gas supply system which requires ultrapure gases.

Furthermore, in the apparatus shown in Fig. 31 (a), when supplying process gas from process gas supply piping line 602 (Fig. 31 (e)), process gas supply piping lines 638 and 639 of the process gas supply piping lines 603 and 604 will become the dead zone for the gas, and hence no exchange of gas can take place, causing the reduction of the purity of the process gas supplied. Moreover, when supplying process gas from the process gas supply piping line 604, not only the piping lines 628 and 642, which are used for exhaust discharge of purge gas and vacuum exhaust discharge of the gas pipelines, but the process gas supply piping lines 638, 639, 645, and 646 are also closed; therefore, the pollution inside the pipelines becomes more severe (Fig. 31 (g)). The known technique as described above illustrates the situation wherein there are three process gas supply piping lines; in real installation, the number is much greater, and hence the effect of pollution becomes worse.

Therefore, for a piping system which supplies a number of process gases to one single process apparatus, it is hoped that there is a systematic technique wherein purging and vacuum exhaust discharge can take place independently in each process gas supply pipeline and each piping line of the

separately independent flow path different from the above-mentioned flow path formed in between the other end of each process gas supply piping line mentioned above and the other end of the purge gas piping line mentioned above; at least two valves which are mounted on a number of flow paths different from the above-mentioned flow path formed in between the other end of each process gas supply piping line mentioned above and one end of the process apparatus piping line mentioned above.

As in the first aspect, the second aspect of the present invention is characterized in that the purge gas piping line mentioned above is connected to the exhaust pipeline by means of at least one flow meter and at least one flow control valve.

As in the first and second aspects, the third aspect of the present invention is characterized in that the at least two valves separately provided in independent flow paths mentioned above and the at least two valves provided in a number of flow paths mentioned above are an integrally formed monoblock valve.

As in the third aspect, the fourth aspect of the present invention is characterized in that the number of the above-mentioned monoblock valves is no less than that which is less than that of the process gas supply piping lines mentioned above by one.

1. A device whose major part is made of stainless steel, at least a part of the above-mentioned stainless steel surface exposed herein the internal part of the device being constituted of two layers, a layer whose main component is the oxide of chrome, formed from near the interface of the stainless steel and the passive state film, and a layer whose main component is the oxide of iron, formed near the surface of the passive state film, with the passive state film having a thickness above 50A° and formed by heating oxidation of stainless steel at a temperature of above 150°C but below 400°C.

2. A device whose major part is made of stainless steel, at least a part of the above-mentioned stainless steel surface exposed herein the internal part of the device having a passive state film of a thickness above 100A° formed from a layer whose main component is the mixed oxides of the oxide of chrome and the oxide of iron, the passive state film being formed by heating oxidation of stainless steel at a temperature above 400°C but below 500°C.

3. A device whose major part is made of stainless steel, at least a part of the above-mentioned stainless steel surface exposed herein the internal part of the device having a passive state film of a thickness above 130A° formed from a layer whose main component is the oxide of chrome, the passive state film being formed by heating

embodiment of the piping system of the present invention; Fig. 2 is a fragmentary enlarged lock diagram showing the 4-way treble valve as in the above first preferred embodiment; Fig. 3 is an fragmentary enlarged block diagram showing the 4-way quadruple valve used in the present invention.

Fig. 4 and Fig. 7 are diagrams illustrating the connection of the gas filter pipelines of the gas piping lines of the process apparatus. Fig. 5, Fig. 6, Fig. 8, and Fig. 9 are diagrams illustrating the operation of the gas filter piping as shown in Fig. 4 and Fig. 7.

Fig. 10 through Fig. 28 are diagrams illustrating the operation of the preferred embodiment as shown in Fig. 1.

Fig. 29 is a fragmentary block diagram showing the second preferred embodiment; Fig. 30 is a fragmentary block diagram showing the third preferred embodiment.

Fig. 31 (a) through (g) are block diagrams showing the gas supply piping device of a process apparatus used in the past; Fig. 32 is a diagram showing, in the gas supply piping device for a process apparatus, the test result of the dew point change under the condition that the piping system is closed.

In the following, an embodiment of the present invention will be described in detail with the drawings.

Description of the Apparatus

135, 136, 137, 138, 139, 140, 141, 142, 163, 164, 166, 167, 171, 172, and 176 are stop valves, among these, the combinations of stop valves 111, 114, 120, stop valves 112, 115, 121, stop valves 113, 116, 122, stop valves 123, 126, 132, stop valves 124, 127, 133, and stop valves 125, 128, 134 are monoblock valves which integrate three valves into one and minimize the dead zones; especially valves 114, 115, 116, 126, 127, and 128, they themselves have the function of a bypass line. In addition, the combinations of stop valves 135, 136, 137, 138, and stop valves 139, 140, 141, 142 are monoblock valves which integrate four valves into one and minimize the dead zones. Furthermore, 163 and 164, as well as 166 and 167 are respectively 2-valve integrated 3-way dual valves. 117, 118 and 119 are pressure regulators. 129, 130 and 131 are mass flow controllers, which can also be floater flow meters attached with needle valves. Besides, although it is not shown in Fig. 1, according to need, it is also possible to utilize a 3-valve-integrated monoblock valve to mount the gas filter in the same way as mounting the pressure regulator or the mass flow controller. In the mounting of the gas filter, for one single process gas supply piping system, it is also possible to use one or two units of 4-valve-integrated monoblock valves to mount two gas filters. This will be explained later. 169 and 174 are needle valves, 170 and 175 are floater flow meters,

apparatus, 151 and 161. are integrated into one for exhaust discharge; or a single pipeline can be used for discharging exhaust.

Fig. 2 is an example of the monoblock valve 230 used in this embodiment. Three valves, 223, 226, 232 are integrated into one, and in between valves 223 and 232 is provided with a mass flow controller 229. This monoblock valve 230 does not become the dead zone of the gas, but is a high performance valve that can form a bypass.

Fig. 3 is a diagram of one of the 4-valve-integrated monoblock valves used in the present invention. The piping lines 143 and 144, 145 and 146, 147 and 148, and 149 and 150 of the confluent part of the process gas become extremely small by being made into monoblocks; therefore, the reduction of the purity of the process gas created as a result of the dead zones of the gas within the valves can be suppressed to a minimum. Hence, such monoblock valve is a high performance valve which is characterized in that it can supply 2 kinds of process gas independently to 2 piping lines of the process apparatus with no reduction of the purity of the highly pure process gas.

A connection example showing the state of connecting filters

Furthermore, by means of combining these two kinds of

purging gas filter 211, open valves 204, 207, 208, 209, close valves 205, 206, heat gas filter 211 with a heater or the like. At this time the flow of gas is as shown in Fig. 5. Further, when using gas filter 211 and purging gas filter 210, open valves 205, 206, 208, 209, close valves 204, 207, heat gas filter 210 with a heater or the like. At this time the flow of gas is as shown in Fig. 6. With such method, as the regeneration of the gas filter is carried out using the process gas, although it is not necessary to consider the substitution of gas within the gas filter, this method cannot be applied when, because of various reasons, the flow of process gas cannot be supplied in a large quantity.

Following, a connection example when regenerated gas is separately supplied is illustrated with reference to Fig. 7 through Fig. 9.

Fig. 7 is a connection example of the above-mentioned piping system. 301 is a pipeline from the process gas supply piping line, which is connected to valves 111, 112, 113, 123, 124, 125 of the 3-valve-integrated monoblock valves shown in Fig. 1. 302 is a piping line leading to the process apparatus, which is connected to valves 120, 121, 132, 133, 134 of the 3-valve-integrated monoblock valve shown in Fig. 1. 303 is a purge gas supply pipeline, which is connected to the exhaust duct via the needle valve and

gas (e.g. Ar, etc.) when the process gas is not used. The flow of the gas is represented by thick lines. When the apparatus has been installed, the dew point of the gas system is high. To decrease the water moisture within the wall of the piping earlier, as shown in Fig. 10, in the state wherein valves 108, 109, 110, 111, 112, 113, 114, 115, 116, 120, 121, 122, 123, 124, 125, 126, 127, 128, 132, 133, 134, 135, 138, 139, 142, 164, 167, 171, 172, and 176 are all open, and valves 114, 137, 140, 141, 163, and 166 are all close, using needle valves 169 and 174 to regulate the flow rate of each process gas to reach a degree of 20 l/min, supply the purge gas (e.g. Ar etc.) from process gas supply pipine lines 102, 103, and 104 to the reaction chamber of the apparatus; abundant gas is used to carry out the purging within the piping system. By means of this, when the dew point reaches a degree of -80°C, for instance, in the state wherein valves 108, 109, 110, 111, 112, 113, 120, 121, 122, 123, 124, 125, 132, 133, 134, 135, 138, 139, 142, 164, 167, 171, 172, and 176 are all open, and valves 114, 115, 116, 126, 127, 128, 136, 137, 140, 141, 163, and 166 are all close, using needle valves 169 and 174 to regulate the flow rate, supply purge gas (e.g. Ar, etc.) from process gas supply piping lines 102, 103, and 104 to the front of the reaction chamber of the apparatus. Then proceed with the purging of the pressure regulators, mass flow controllers

can also take place in the state wherein valves 136, 137, 140 and 141 are all open, and valves 135, 138, 139, and 142 are all close; valves 135, 138, 140, and 141 are all open, valves 136, 137, 139, and 142 are all close; or valves 136, 137, 139, and 142 are all open, valves 135, 138, 140, and 141 are all close.

Following, using Fig. 12 through Fig. 23, the supply of process gas is explained. The thick lines represent the pipelines wherein gases flow.

First of all, the operation of the supply of one kind of gas to the reaction chamber 101 of the process apparatus is illustrated using Fig. 12 through Fig. 17.

Firstly, the way of supplying process gas from the process gas supply piping line 102 is explained. In order to supply highly pure process gases to the process apparatus, substitution of process gas for residual purge gas (e.g. Ar) in the supplying pipelines is carried out. Just before that, in the state as shown in Fig. 11, regulate the flow rate of purge gas (e.g. Ar) supplied from the process gas supply piping lines 102, 103, and 104 by means of needle valves 169, 174, and supply the gas to the reaction chamber of the process apparatus, so that the entire gas system is under purge.

Subsequently, close valves 135, stop the supply of purge gas from process gas supply piping line 102, then

reaction chamber 101 of the apparatus. The flow of gas at this time is as shown in Fig. 13.

Following, in respect to the way of supplying gas from the process gas supply piping line 103, an illustration is given. In order to supply highly pure process gas to the process apparatus, similar to the method of supplying gas as mentioned above, the operation of substituting process gas for residual purge gas (e.g. Ar) in the supplying pipelines is being carried out. Just before that, in the state as shown in Fig. 11, regulate the flow rate of purge gas (e.g. Ar) supplied from the process gas supply piping lines 102, 103, and 104 by means of needle valves 169, 174, and supply the gas to the reaction chamber of the process apparatus, so that the entire gas system is under purge. Close valves 139, stop the supply of purge gas from process gas supply piping line 103, then close valves 171, 167, 172, and 176, stop the purging from the purge gas exhaust pipelines 105, 106. Next, close valves 138, 142, open valves 140, 172, 176, start again the purging of the process gas supply piping line systems 102, 104. At this time the flow of gas is as shown in Fig. 14. Then, open valves 115, 127, 138, 141, 164, 166, use vacuum exhaust pipeline 107 to proceed with the vacuum exhaust discharge of the process gas supply piping line 103 and the piping lines of the process apparatus. After the degree of vacuum in the piping lines has reached,

in Fig. 11, regulate the flow rate of purge gas (e.g. Ar) supplied from the process gas supply piping lines 102, 103, and 104 by means of needle valves 169, 174, and supply the gas to the reaction chamber of the process apparatus, so that the entire gas system is under purge. Close valves 171, stop the supply of purge gas the from the process gas supply piping line 104, then close valves 167, 172, 176, stop the purging from purge gas exhaust pipelines 105, 106. At this time the flow of gas is as shown in Fig. 16. Then, open valves 116, 128, 166, use vacuum exhaust pipeline 107 to proceed with the vacuum exhaust discharge of the process gas supply piping line 104 and the piping lines of the process apparatus. After the degree of vacuum in the piping lines has reached, say, 1×10^{-2} Torr, close valve 166, supply process gas from the process gas supply piping line 104 to fill the piping system with process gas. After the piping system is filled with process gas, stop the supply of process gas, and proceed with the vacuum exhaust discharge in the piping system in the same way as the vacuum exhaust discharge of purge gas. This supplying of process gas from the process gas supply piping line 104 and the vacuum exhaust discharge of the piping lines are usually repeated over 5 times. After thorough substitution of purge gas and process gas in the piping system has taken place, in the state wherein valves 116, 128, 164, 166 are close, and

exhaust pipelines 105, 106. Next, close valves 135, 139, 142, open valves 140, 172, 176, start again the purging of the process gas supply piping line system 104. At this time the flow of gas is as shown in Fig. 18. Then, open valves 114, 115, 126, 127, 137, 141, 166, use vacuum exhaust pipeline 107 to proceed with the vacuum exhaust discharge of the process gas supply piping lines 102, 103 and the piping lines of the process apparatus. After the degree of vacuum in the piping lines has reached, say, 1 X 10^{-2} Torr, close valve 166, 138 (or 137), supply process gas from the process gas supply piping lines 102, 103 to fill the piping system with process gas. After the piping system is filled with process gas, stop the supply of process gas, and proceed with the vacuum exhaust discharge in the piping system in the same way as the vacuum exhaust discharge of purge gas. This supplying of process gas from the process gas supply piping lines 102, 103 and the vacuum exhaust discharge of the piping lines are usually repeated over 5 times. After thorough substitution of purge gas and process gas in the piping system has taken place, in the state wherein valves 114, 115, 126, 127, 164, 166 are close, and valves 108, 109, 111, 112, 120, 121, 123, 124, 132, 133, 137, 138, 141, 163 are open, by using pressure regulators 117, 118, and mass flow controllers 129, 130, regulate the supply pressure and the flow rate of the process gas, and

apparatus. After the degree of vacuum in the piping lines has reached, say, 1 X 10^{-2} Torr, close valve 166, 142 (141 can also do), supply process gas from the process gas supply piping lines 102, 104 to fill the piping system with process gas. After the piping system is filled with process gas, stop the supply of process gas, and proceed with the vacuum exhaust discharge in the piping system in the same way as the vacuum exhaust discharge of purge gas. This supplying of process gas from the process gas supply piping lines 102, 104 and the vacuum exhaust discharge of the piping lines are usually repeated over 5 times. After thorough substitution of purge gas and process gas in the piping system has taken place, in the state wherein valves 114, 116, 126, 128, 164, 166 are close, and valves 108, 110, 111, 113, 120, 122, 123, 125, 132, 134, 137, 141, 142, 163 are open, by using pressure regulators 117, 119, and mass flow controllers 129, 131, regulate the supply pressure and the flow rate of the process gas, and supply the process gas to the reaction chamber 101 of the process apparatus. The flow of gas at this time is as shown in Fig. 21.

Following, the way of supplying simultaneously process gas supplied from the process gas supply piping lines 103, 104 to the reaction chamber 101 of the process apparatus is illustrated. In order to supply highly pure process gases to the process apparatus, the operation of substituting

times. After thorough substitution of purge gas and process gas in the piping system has taken place, in the state wherein valves 115, 116, 127, 128, 164, 166 are close, and valves 109, 110, 112, 113, 121, 122, 124, 125, 133, 134, 138, 141, 142, 163 are open, by using pressure regulators 118, 119, and mass flow controllers 130, 131, regulate the supply pressure and the flow rate of the process gas, and supply the process gas to the reaction chamber 101 of the process apparatus. The flow of gas at this time is as shown in Fig. 23.

The above is a description of the method of supplying one or two kinds of process gas. As for 3 kinds of process gas, they can also be supplied. Furthermore, when supplying process gas as mentioned above, the individual process gas supply piping line of the process apparatus, except during the opening or closing of directional control valves in the purge gas piping lines, will have no residence of gas.

(Stopping the supply of process gas)

Next, the method of stopping the supply of process gas is illustrated with reference to Fig. 24 through Fig. 28. The operation is similar to that of supplying process gas, only that this is not substituting purge gas with process gas, but substituting process gas with purge gas.

First of all, in respect to the method of stopping the use of process gas supplied from the process gas supply

exhaust discharge of process gas. This supplying of purge gas from the process gas supply piping line 102 and the vacuum exhaust discharge of the piping lines are usually repeated over 5 times. After thorough substitution of purge gas and process gas in the piping system has taken place, close valve 166, supply purge gas from process gas supply piping line 102, open valves 167, 171, regualte the flow rate by means of needle valve 169, by introducing purge gas into purge gas exhaust pipeline 106, proceed with the purging in the system of the process gas supply piping line 102. By means of the above, purging in the unused system of the process gas supply piping lines 103, 104 will not be interrupted; therefore, the supply of process gas can be stopped and purging can be started again in the system of the process gas supply piping line 102. The flow of gas at this time is as shown in Fig. 25. Furthermore, the way of supplying process gas from the process gas supply piping lines 103, 104 can be carried out in the same way; by stopping the supply of process gas, there is no need to interrupt the purging of other unused gas system, the purge can be started again.

Subsequently, in respect to the method of stopping under the circumstance that two kinds of process gas are being simultaneously supplied, an explanation is given. The gas system is in the state wherein valves 108, 109, 110,

gas supply piping lines 102, 103, and the vacuum exhaust discharge of the piping lines are usually repeated over 5 times. After thorough substitution of purge gas and process gas in the piping system has taken place, close valve 166, supply purge gas from the process gas supply piping lines 102, 103, close valves 136, 137, open valves 135, 167, 171, regualte the flow rate by means of needle valves 169, 174, by introducing purge gas into the purge gas exhaust pipelines 105, 106, proceed with the purging in the systems of the process gas supply piping lines 102, 103. By means of the above, without the interruption of the purging in the process gas supply piping line 104, the supply of process gas can be stopped and purging can be started again in the systems of the process gas supply piping lines 102, 103. The flow of gas at this time is as shown in Fig. 27. Further, when purging restarts, close valves 135, 138; valves 136, 167, 171 can also be opened. The flow of gas at this time is as shown in Fig. 28. Similarly, in the process gas supply piping lines 102 and 104, 103 and 104, under the condition that 3 kinds of process gas are simultaneously supplied, stopping the supply of process gas, purging can recommence without the need to interrupt the purging in other unused systems.

(Second and third embodiments)

The first embodiment of the above shows, for

supplying ultrapure process gas of the process gas supply system of the present invention, the external leakage must be suppressed to below 1 X 10^{-11} Torr l/sec; after actual assembly of the pipelines, the external leakage of the piping system must be examined. Normally, the examination of such small leakage uses He leak detector, and with a port for He leak detector formed at a portion of the pipeline. In the preferred embodiment described, by forming a port for the leakage examination in the vacuum exhaust discharge and purge piping line 165, the problem of pollution of the process gas supplied to the reaction chamber of the process apparatus can be solved.

Further, as in the present invention, by utilizing monoblock valves integrated of a number of valves, it not only is effective to enhancing the performance of the gas piping system, but also achieves outstanding effect of the miniaturization of the apparatus. Furthermore, through the use of monoblock valves, the design of the gas supply system, which requires expert knowledge in the past, has become very easy, and there is no gas dead zones; and a highly efficient gas supply piping device for a process apparatus wherein all the process gas supply systems are capable of independent purging and vacuum exhaust discharge can be designed in a simple way.

The possibility of industrial utilization

What is claimed is:

(1) A gas supply piping device for a process apparatus comprising: at least two process gas supply piping lines with one end connected to the gas supply source; a purge gas piping line, with one end connected to the purge gas exhaust discharge system and the other end joined to the other end of said process gas supply piping line by means of a valve; and a process apparatus piping line, with one end connected to each said process gas supply piping line and the other end connected, by means of a valve, to the process apparatus which treats process gas from said gas supply source; and being characterized in comprising: at least two valves which are mounted on a separately independent flow path different from said flow path formed in between the other end of each said process gas supply piping line and the other end of said purge gas piping line; at least two valves which are mounted on a number of flow paths different from said flow path formed in between the other end of each said process gas supply piping line and one end of said process apparatus piping line.

(2) A gas supply piping device for a process apparatus as claimed in Claim 1, wherein said device is characterized in that said purge gas piping line is connected to the exhaust pipeline by means of at least one flow meter and at least one flow control valve.

ABSTRACT OF THE DISCLOSURE

(1) A gas supply piping device for a process apparatus comprising: at least two process gas supply piping lines with one end connected to the gas supply source; a purge gas piping line, with one end connected to the purge gas exhaust discharge system and the other end joined to the other end of said process gas supply piping line by means of a valve; and a process apparatus piping line, with one end connected to each said process gas supply piping line and the other end connected, by means of a valve, to the process apparatus which treats process gas from said gas supply source; and being characterized in comprising: at least two valves which are mounted on a separately independent flow path different from said flow path formed in between the other end of each said process gas supply piping line and the other end of said purge gas piping line; at least two valves which are mounted on a number of flow paths different from said flow path formed in between the other end of each said process gas supply piping line and one end of said process apparatus piping line.

(2) A gas supply piping device for a process apparatus as claimed in Claim 1, wherein said device is characterized in that said purge gas piping line is connected to the exhaust pipeline by means of at least one flow meter and at least one flow control valve.

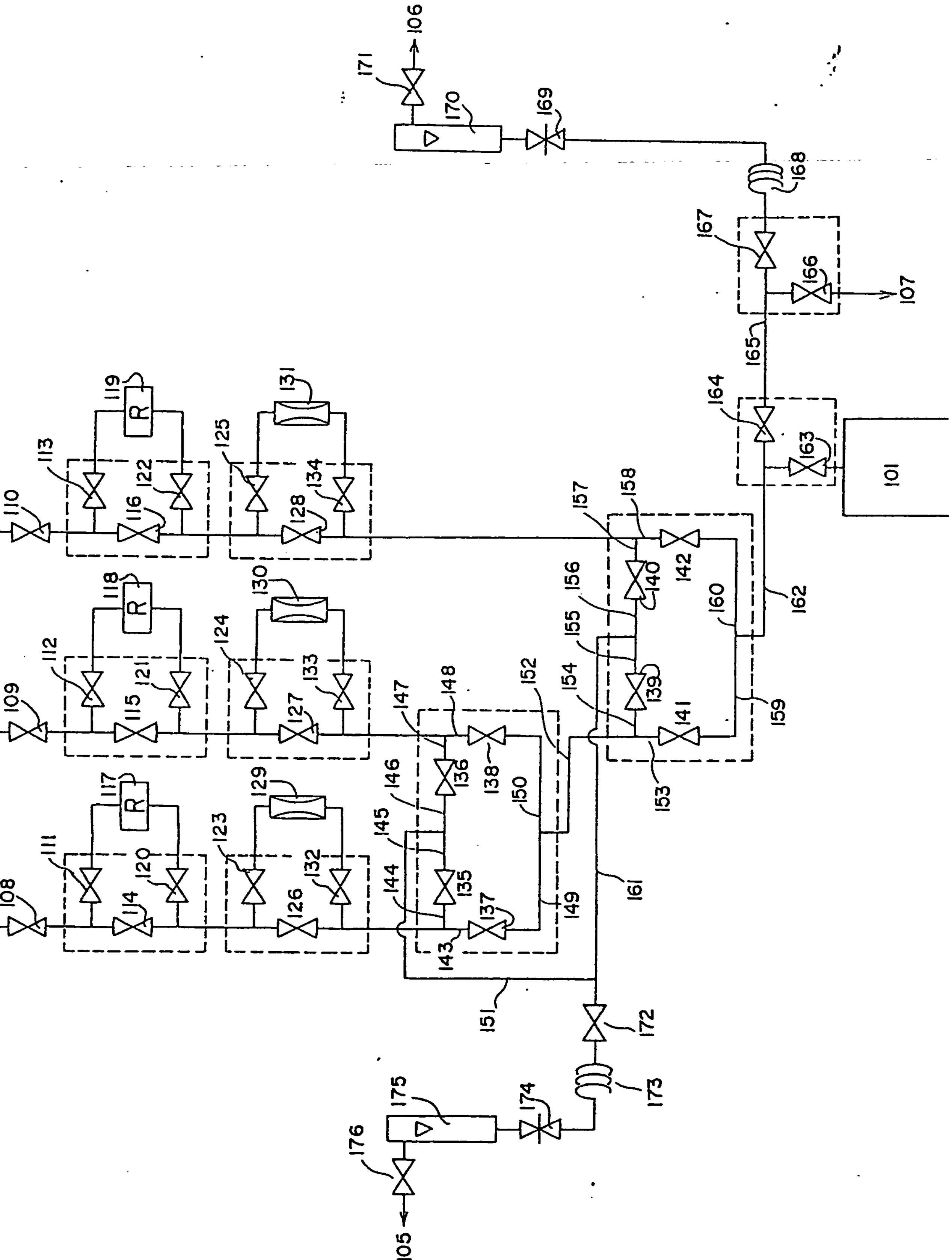
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Fig. 1

Fig. 4

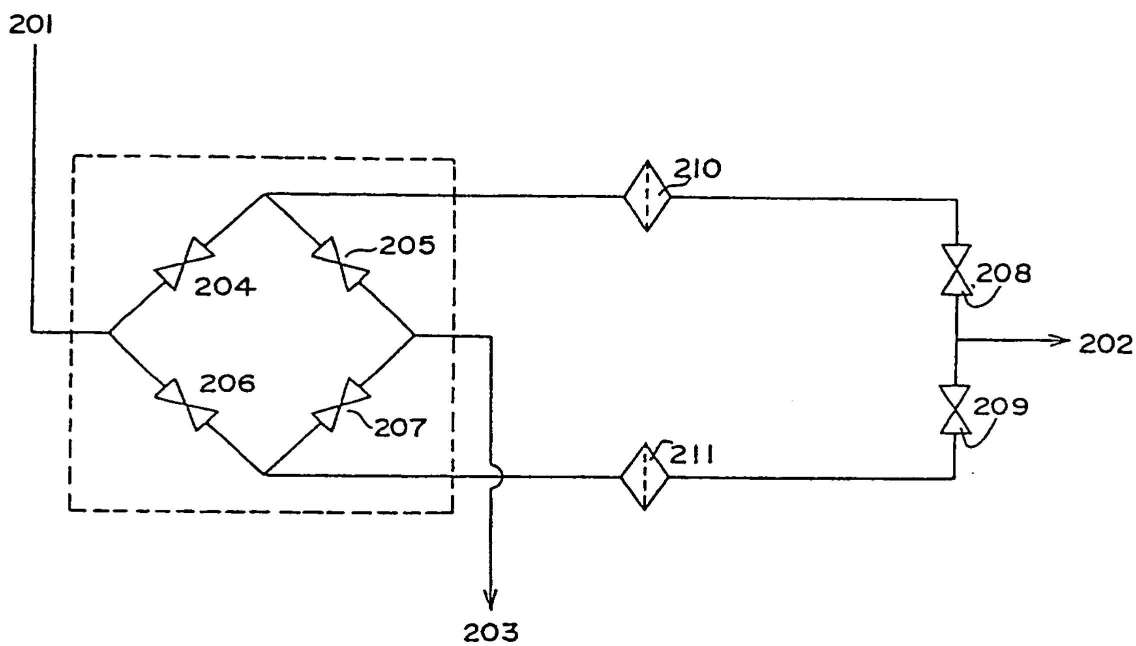


Fig. 6

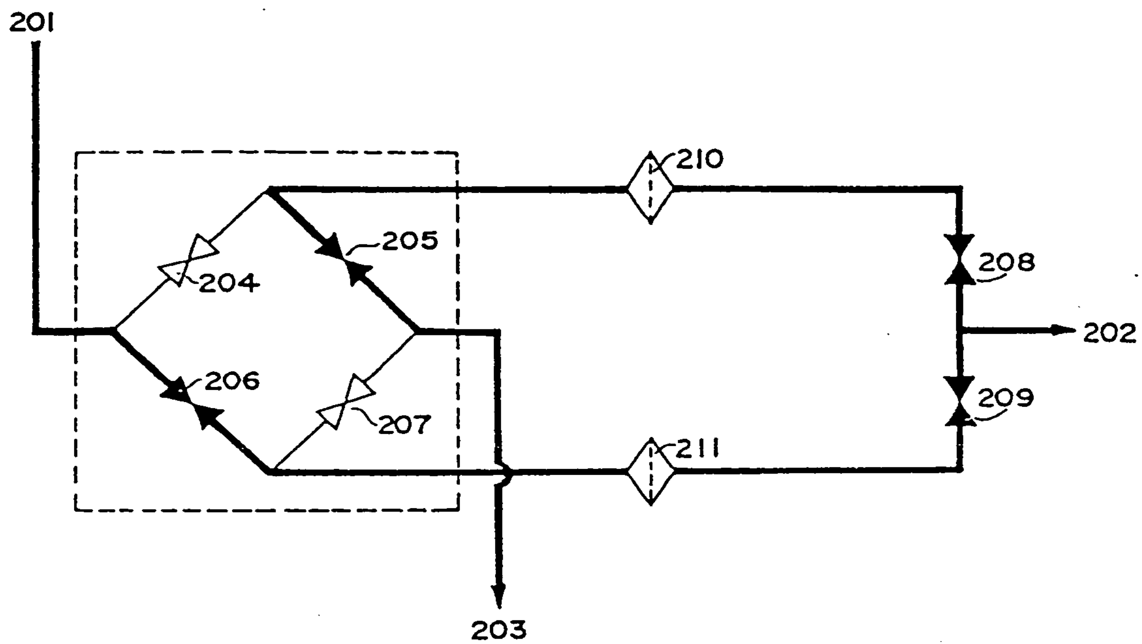
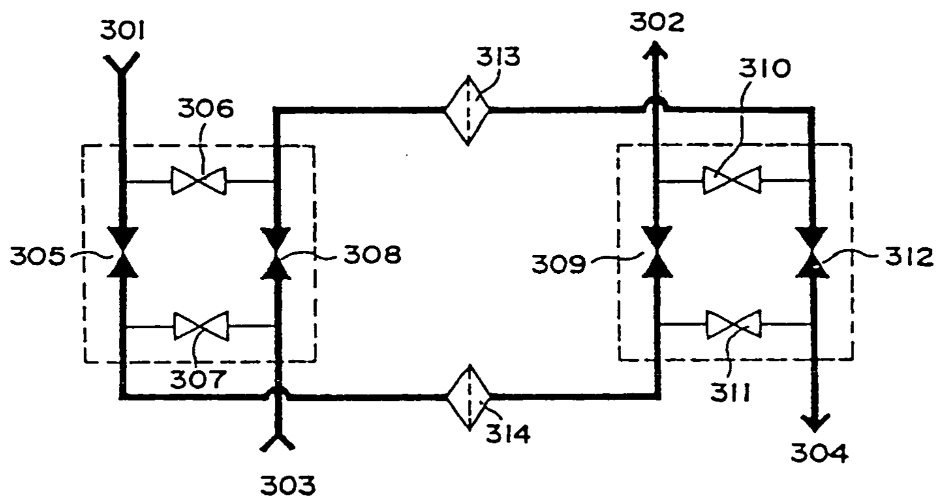


Fig. 8



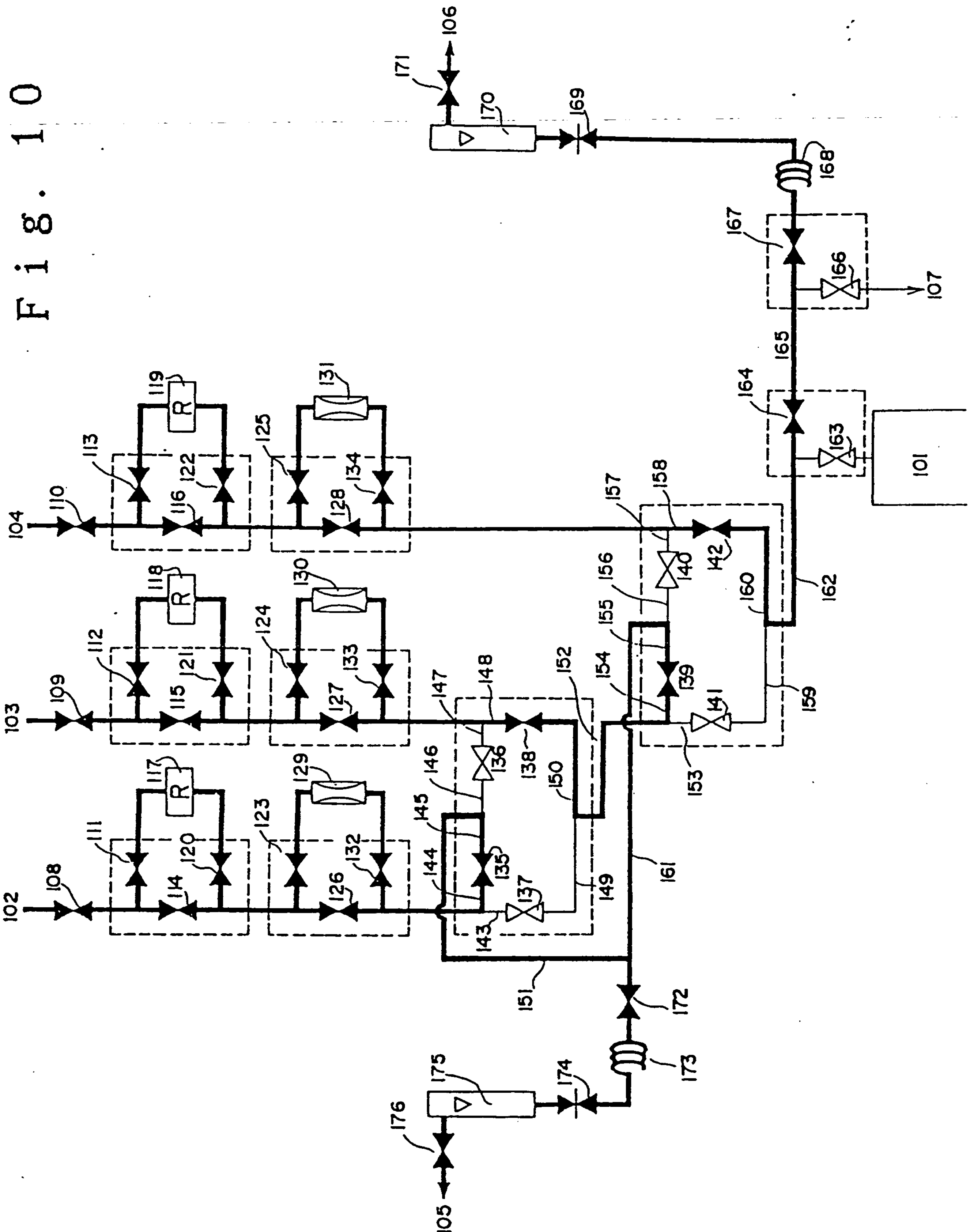

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Fig. 10

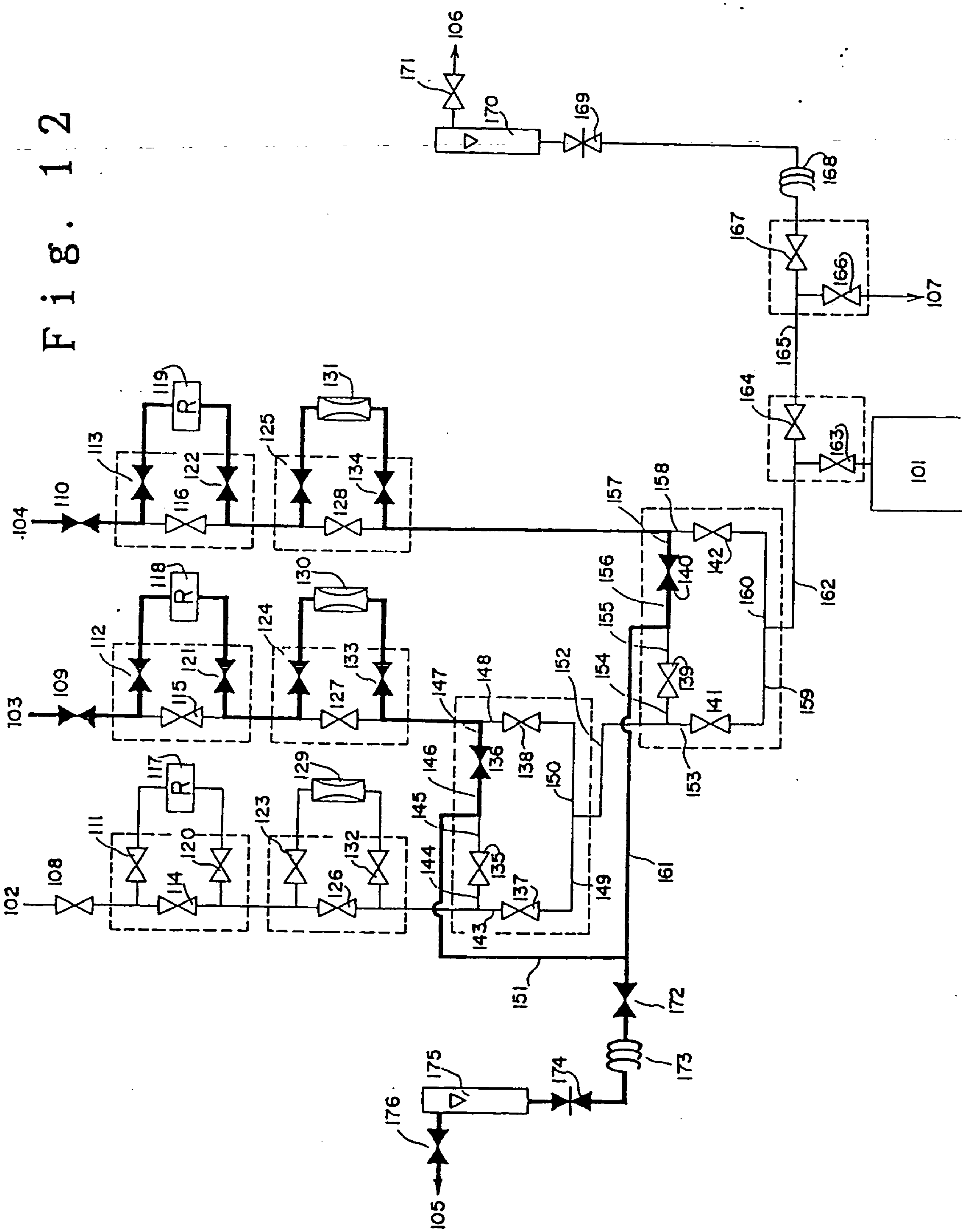
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Fig. 12

Fig. 14

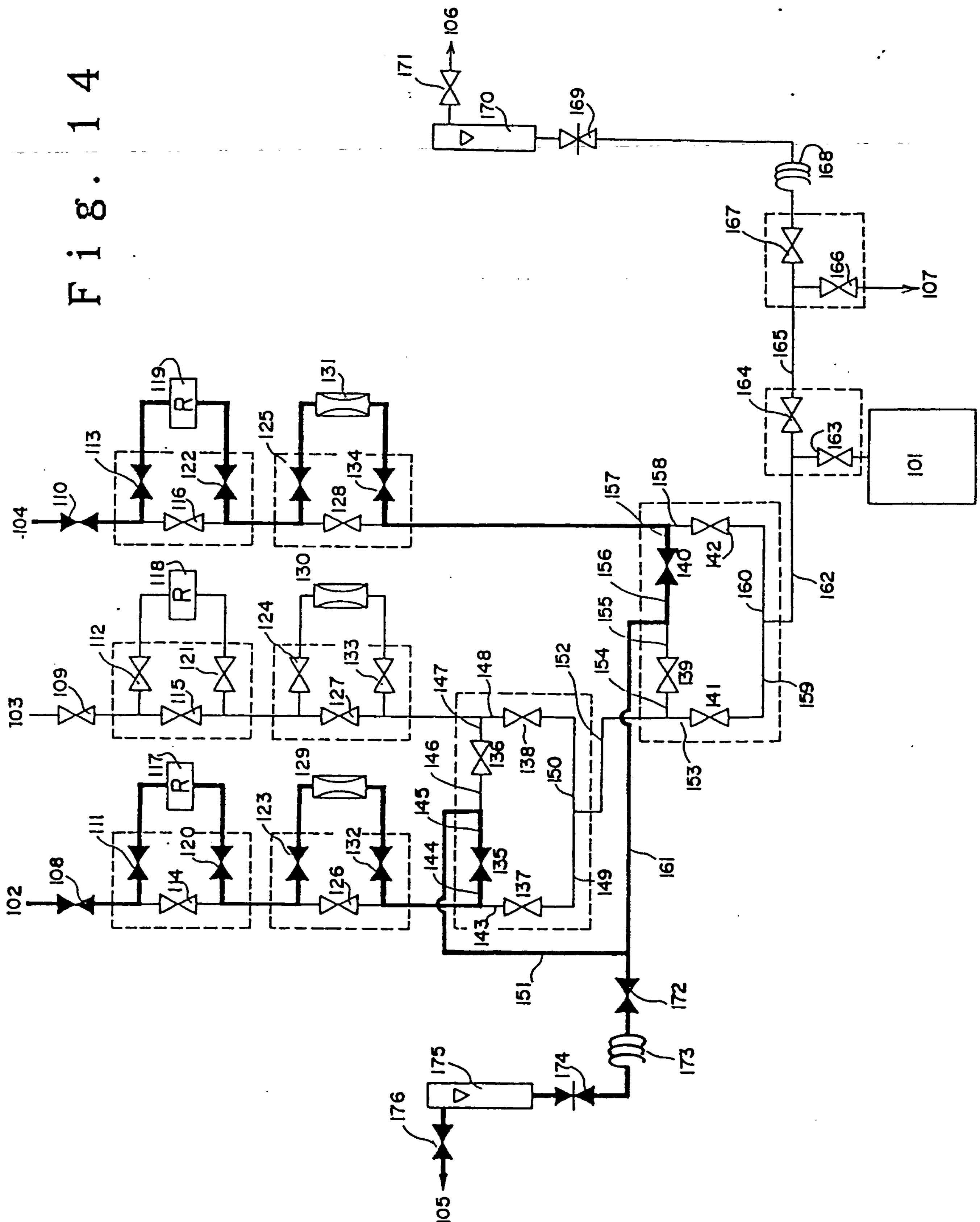
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Fig. 16

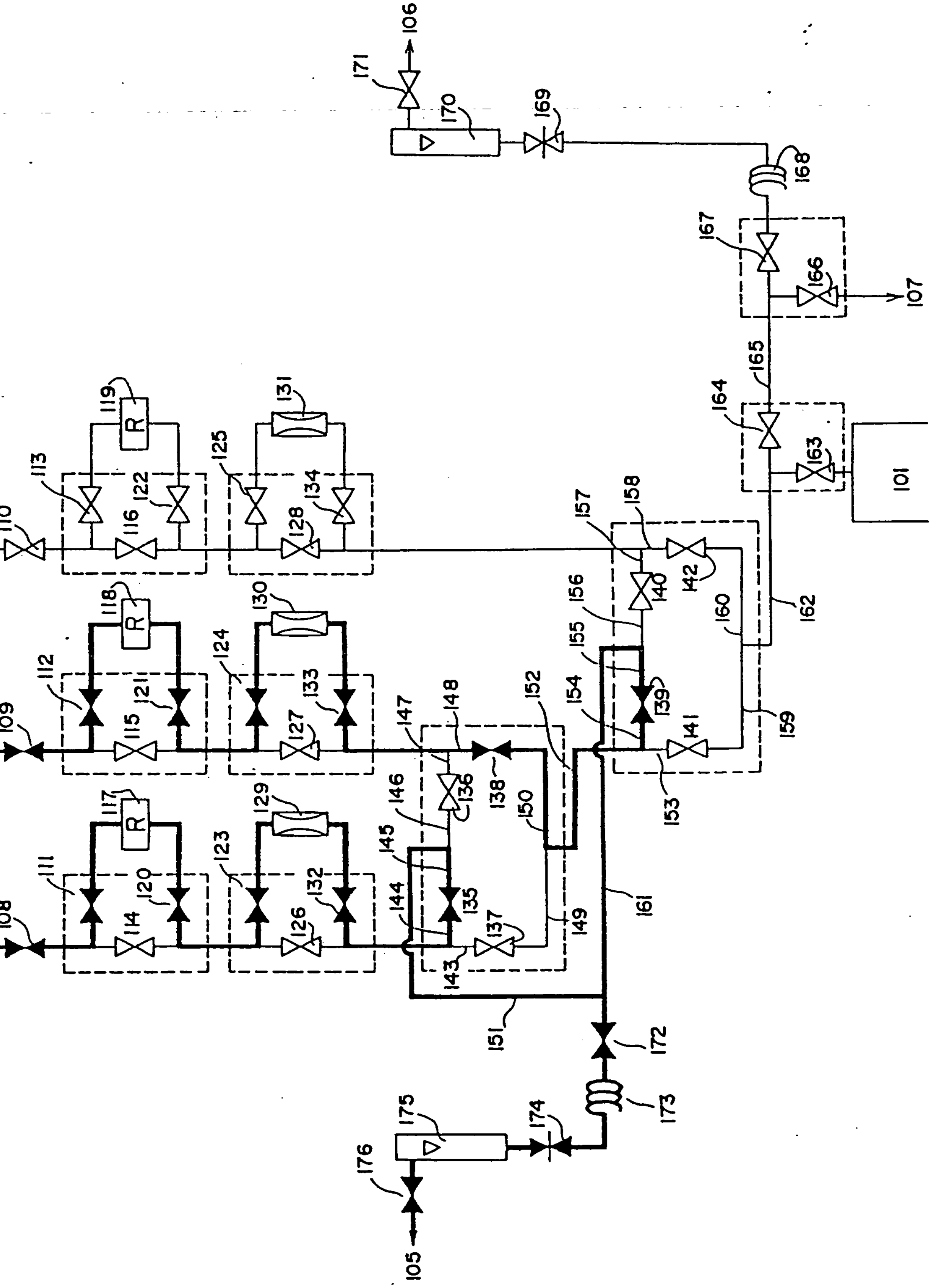
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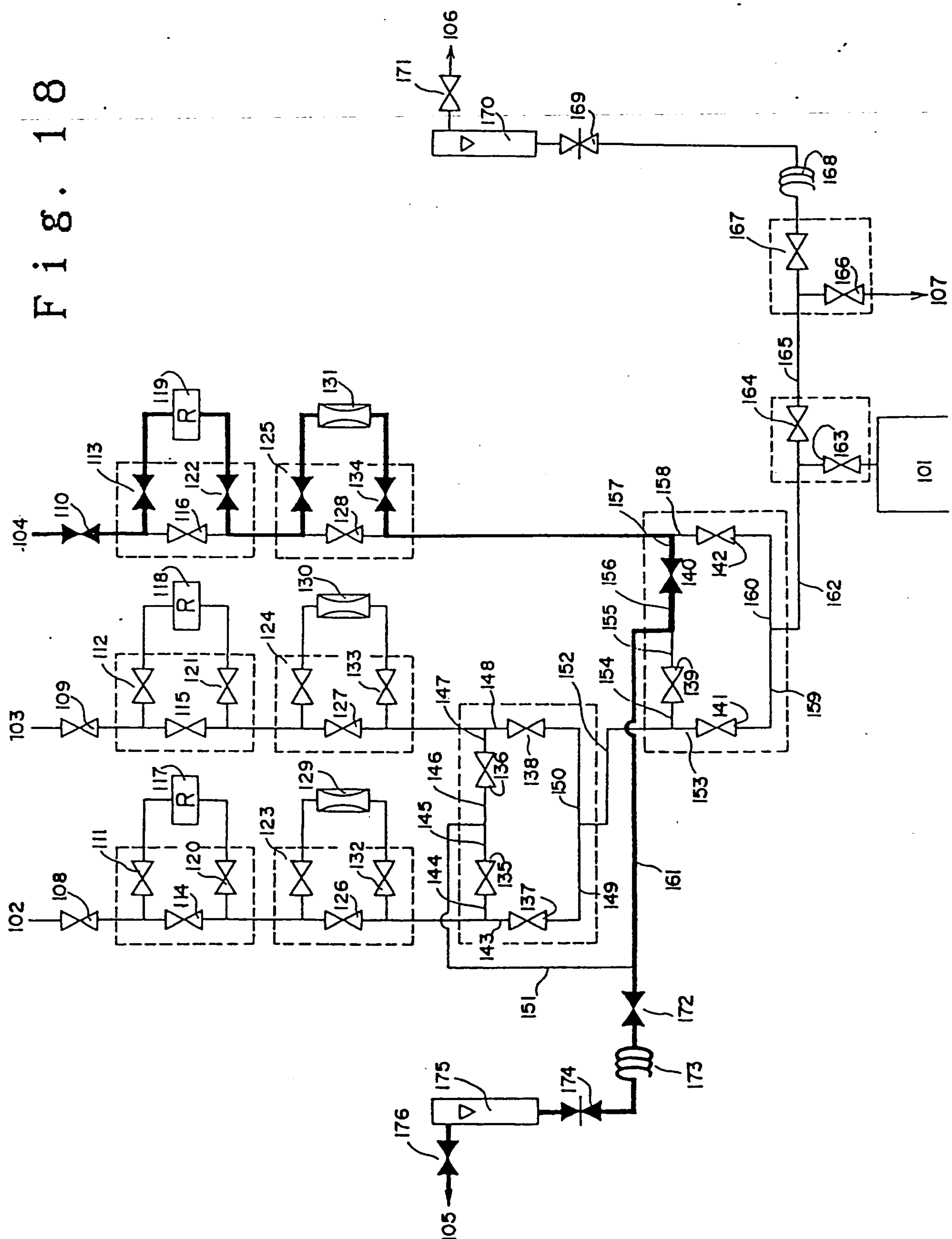
Fig. 18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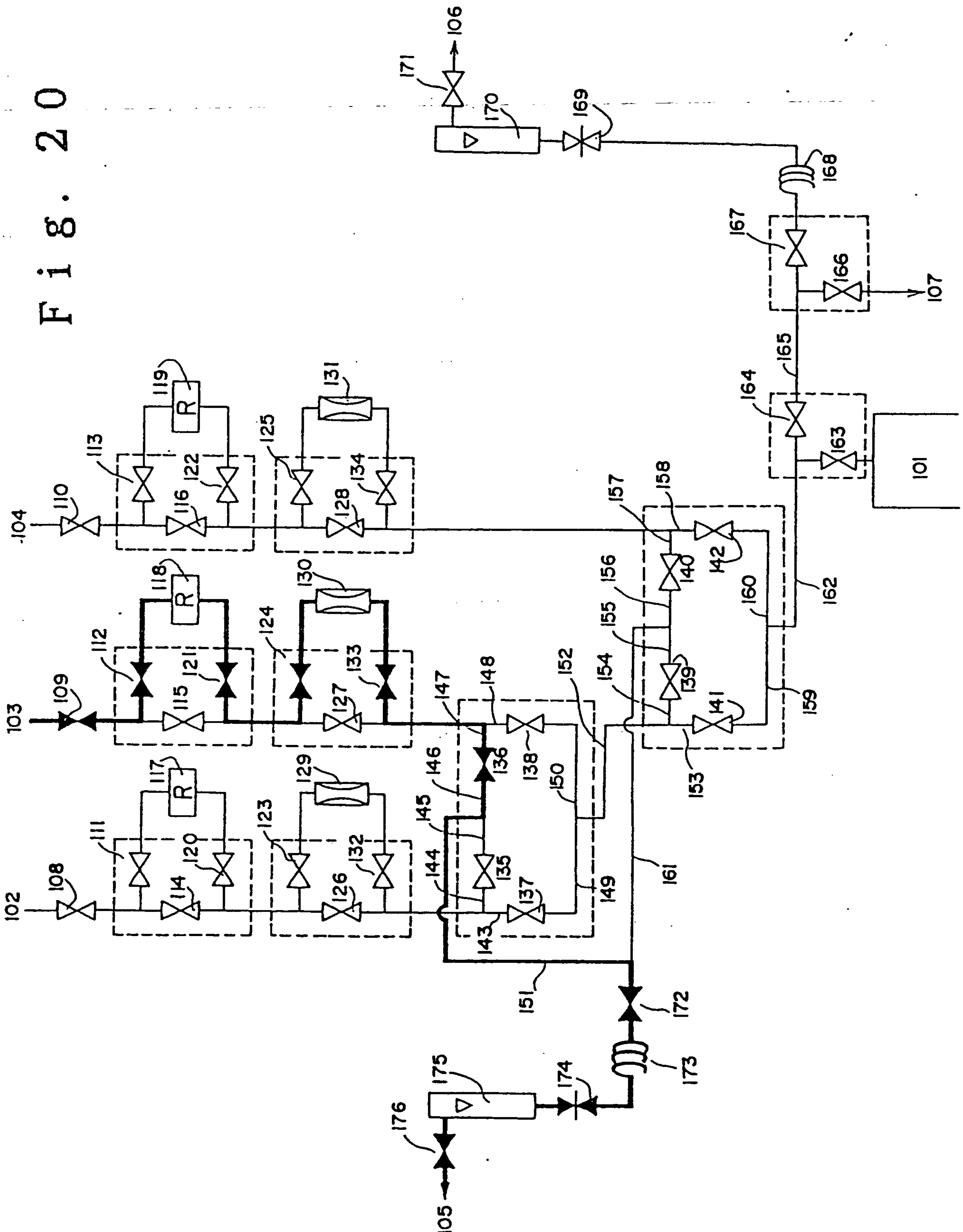
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Fig. 20

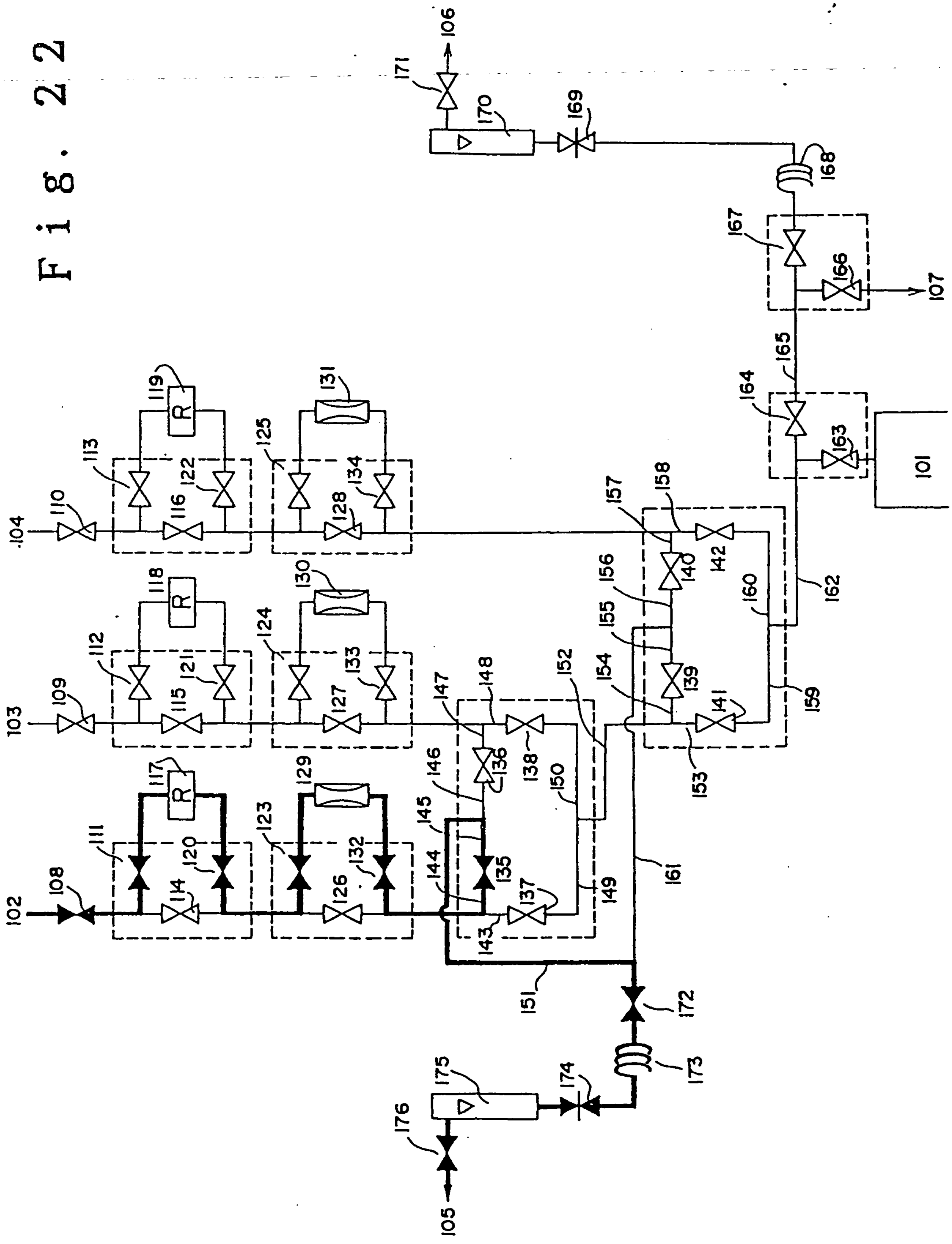
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Fig. 22

Fig. 24

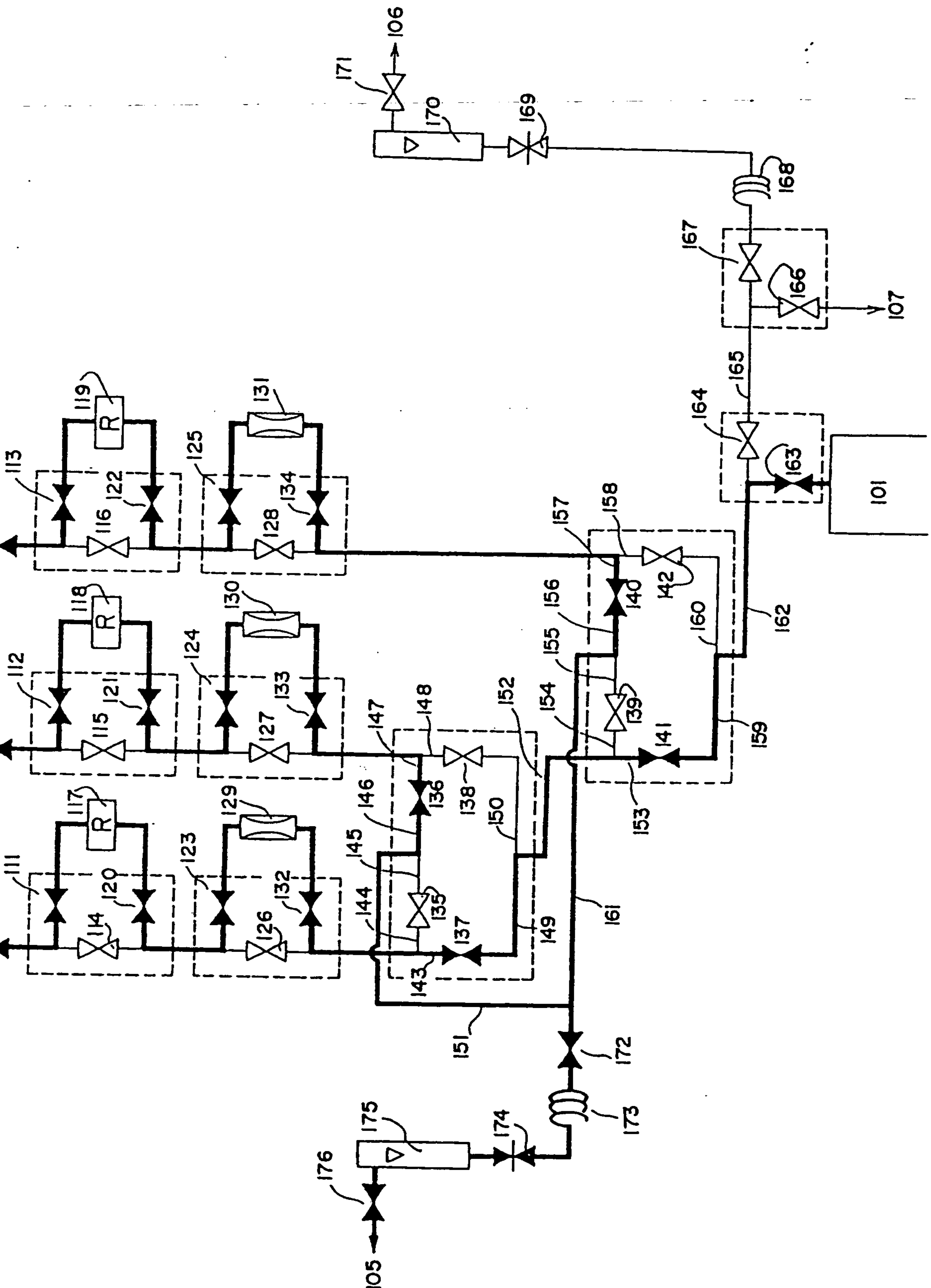
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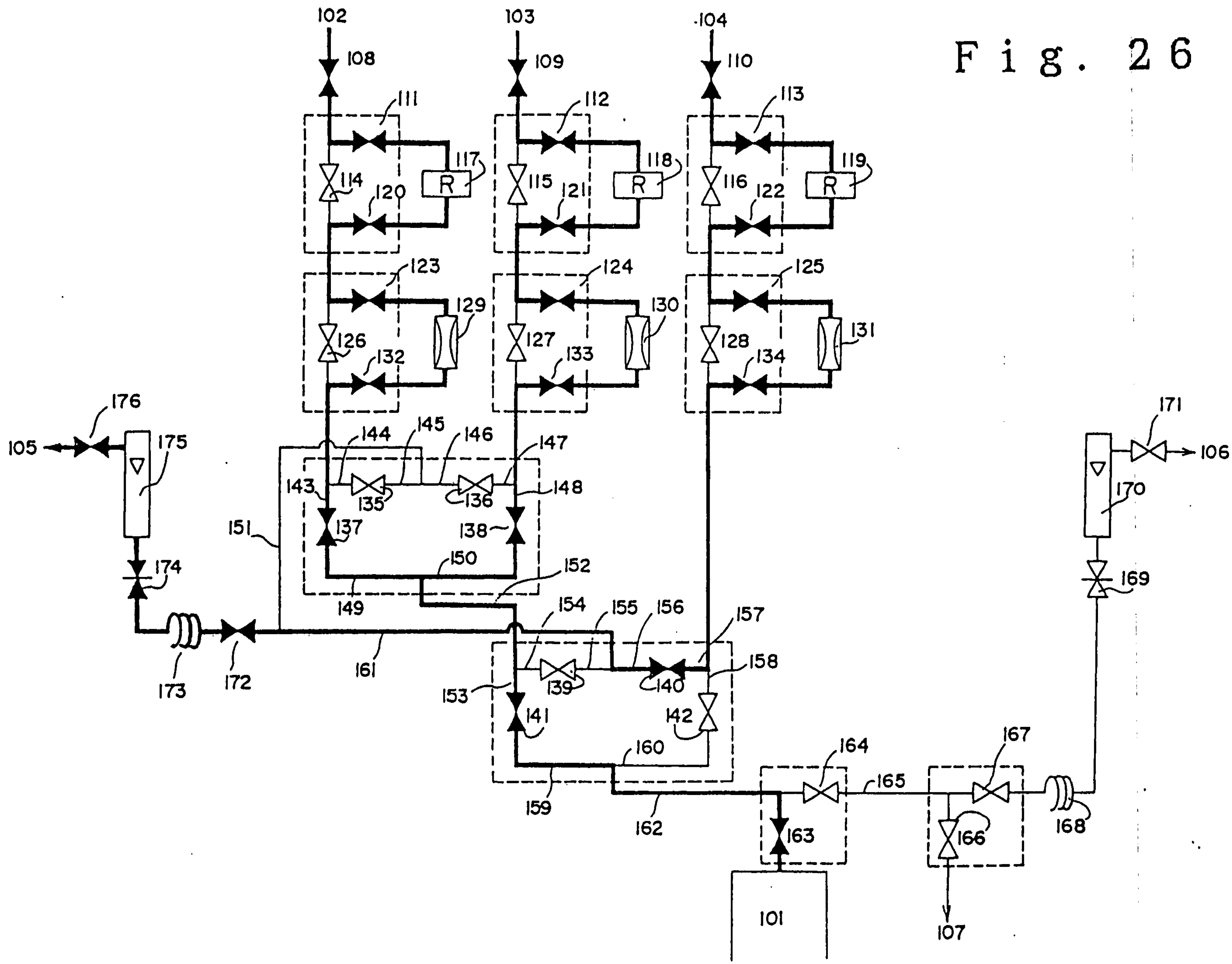
Fig. 26



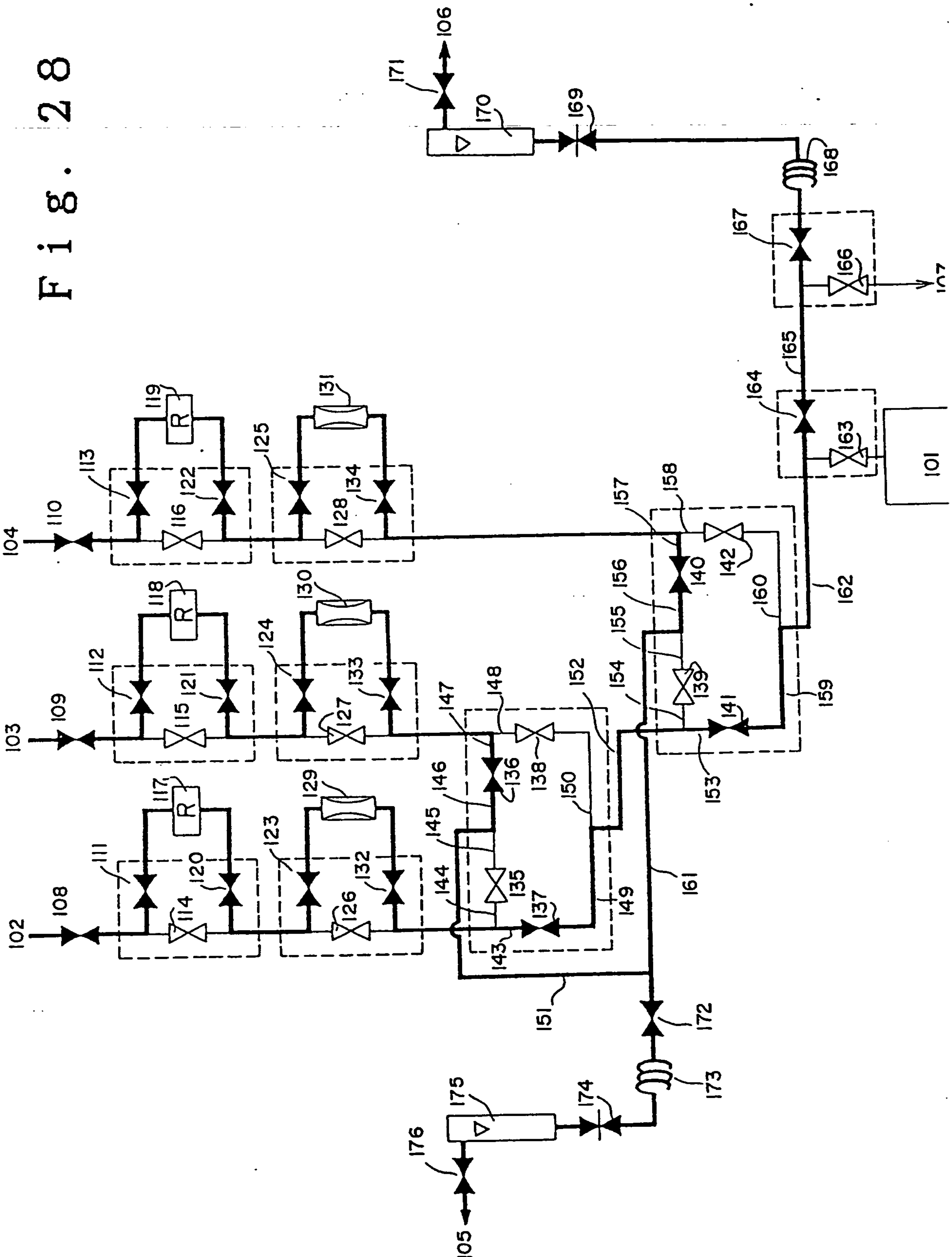

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Fig. 28

Fig. 30

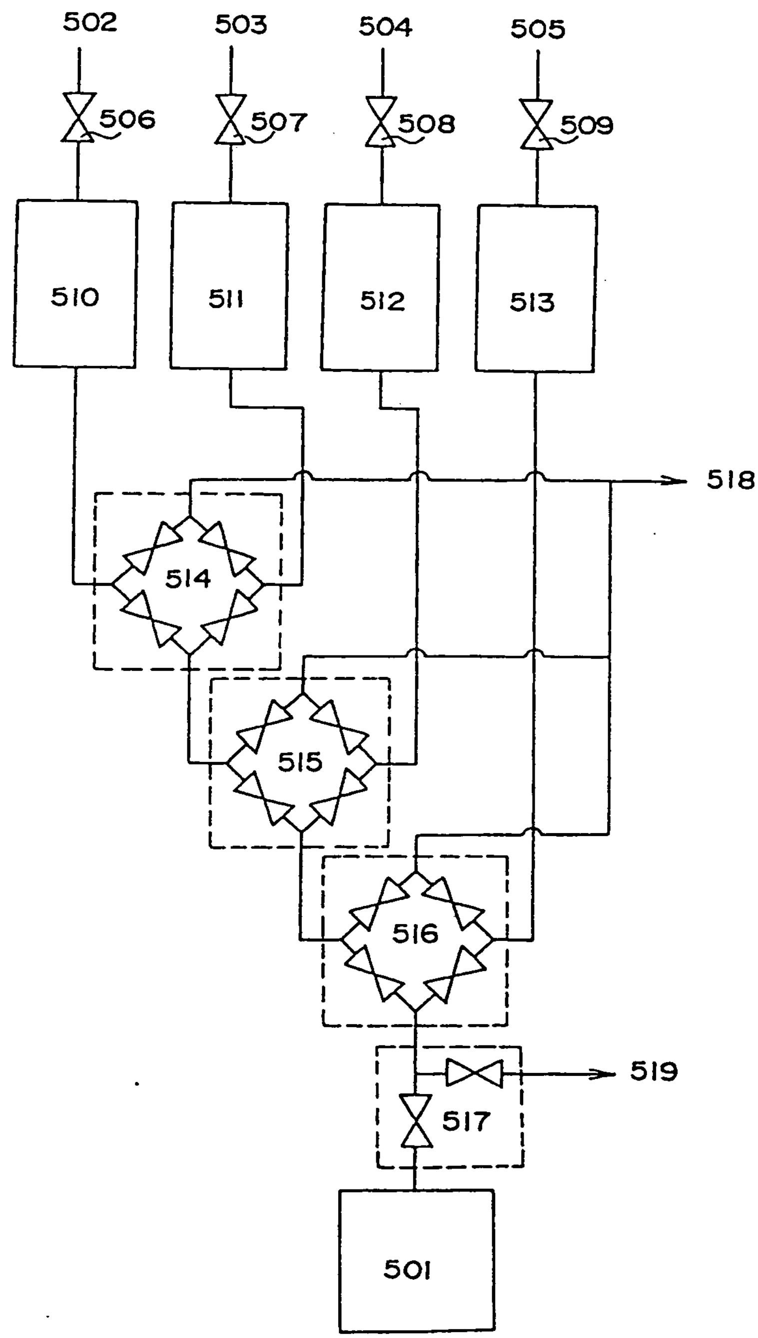
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Fig. 31 (b)

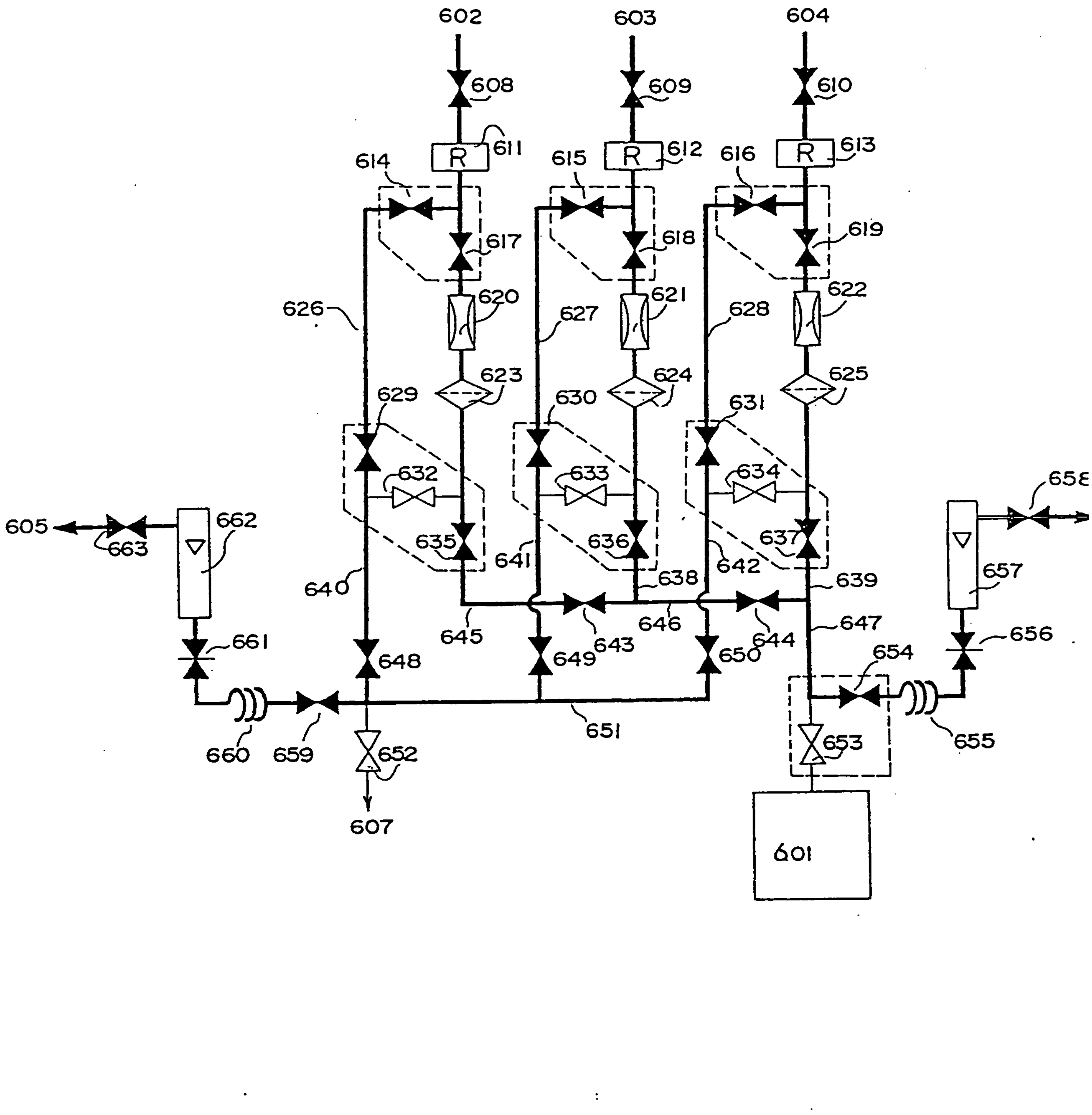
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Fig. 31 (d)

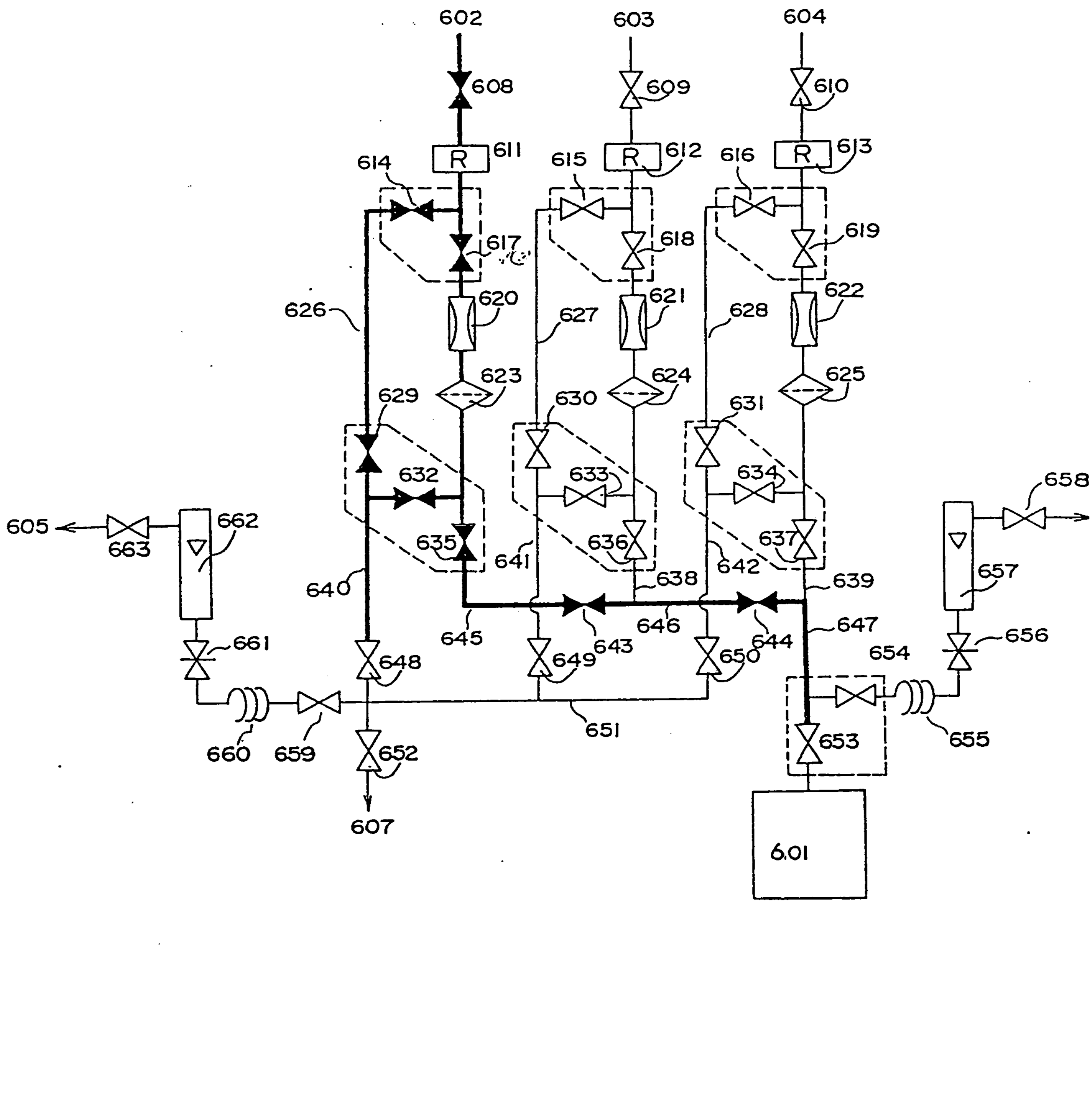
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Fig. 31 (f)

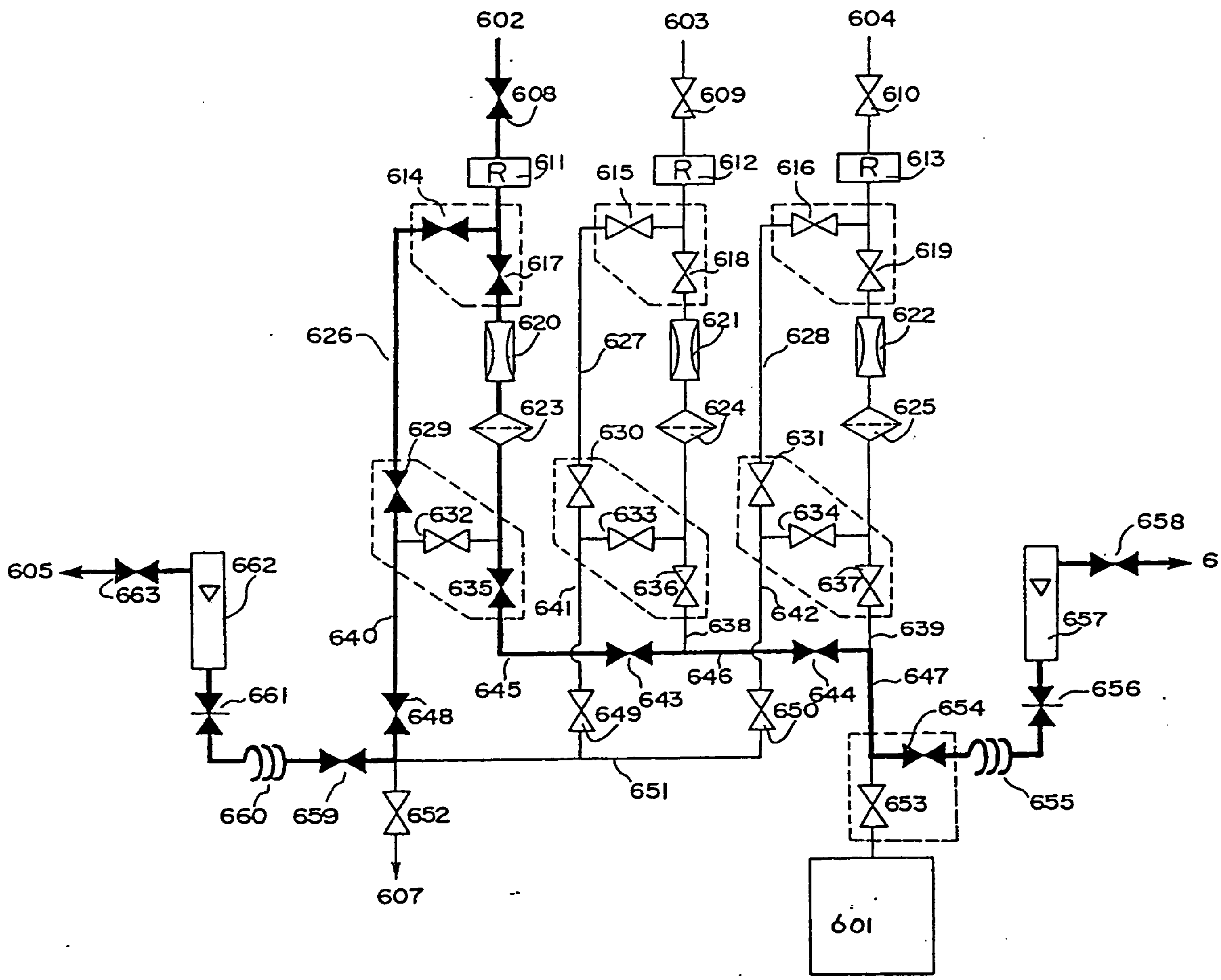
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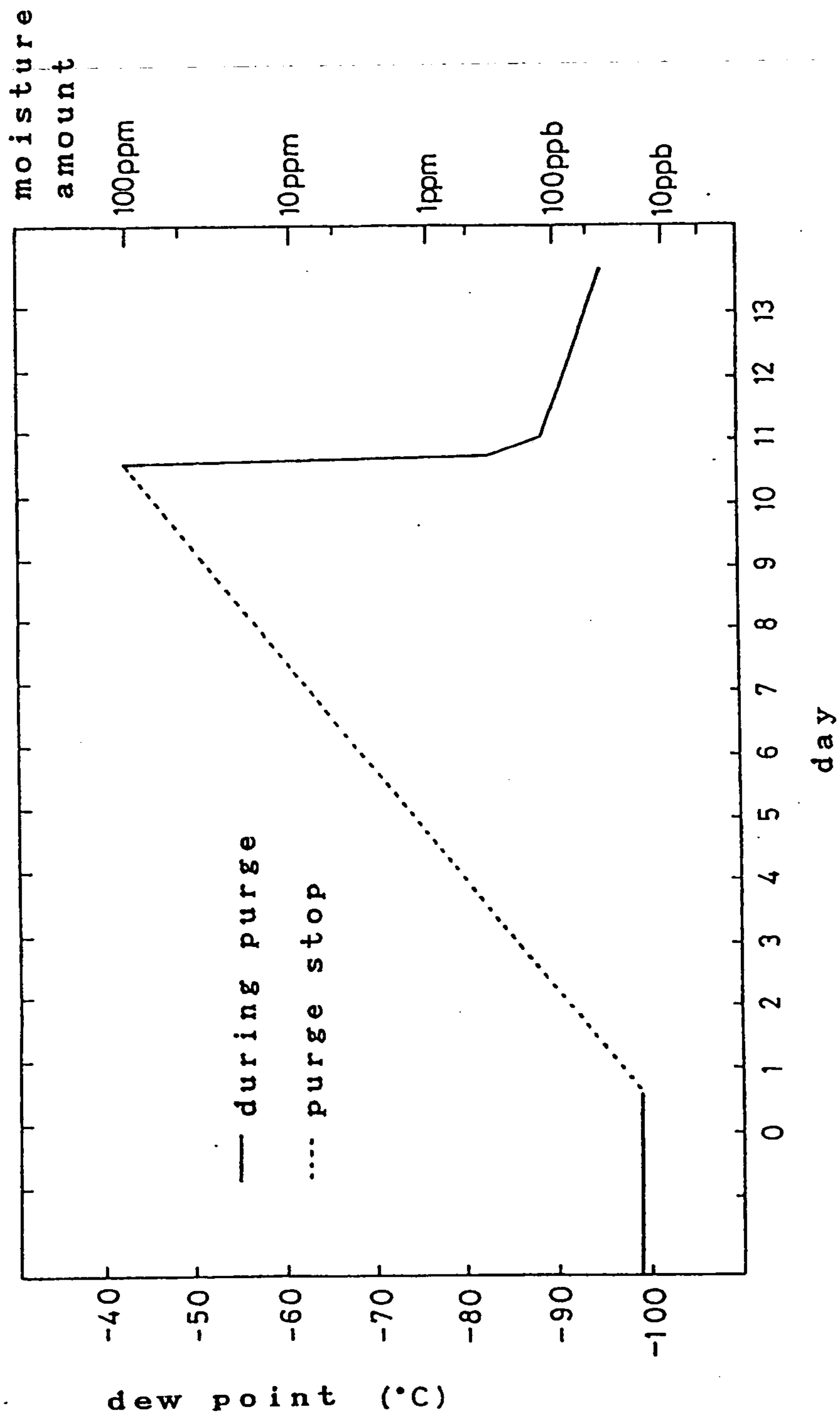
Fig. 32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10ppb
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-50
-60
-70
-80
-90
-100
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— during purge
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